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AIR SAMPLING QUALITY ASSURANCE PROJECT PLAN

Bossert Manufacturing Site
1002 Oswego Street
Utica, New York

Prepared by:

Removal Support Team
Weston Solutions, Inc.
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Prepared for:

U.S. Environmental Protection Agency
Region II - Removal Action Branch
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Approved by:

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Date: 5/14/03

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Quality Assurance Officer

EPA

_____

Date: _____

Jack Harmon
On-Scene Coordinator

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The following elements are provided in the RST Generic Quality Assurance Project Plan (QAPP) and are included by reference:

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1.0 INTRODUCTION

Presented herein is the Air Sampling Quality Assurance Project Plan (QAPP) for the sampling event to be conducted at the Bossert Manufacturing Site by the Region II Removal Support Team (RST). The site QAPP has been developed at the request of the United States Environmental Protection Agency (EPA) in accordance with the RST generic Quality Assurance Project Plan (QAPP).

This plan is based on information currently available and may be modified on site in light of field screening results and other acquired information. All deviations from this QAPP will be noted in the Sampling Trip Report.

2.0 PROJECT DESCRIPTION

Bossert Manufacturing Cooperation is located at 1002 Oswego Street in Utica, Oneida County, New York. It consisted of a former production facility situated on a parcel of land six acres in size. The on-site building was previously demolished. In the past, the site was used for the stamping, welding and fabrication of sheet metal items such as brake backing plates and steel floor grates from 1896 to the 1980s. As a result of manufacturing practices and salvage operations performed subsequent to plant closure, the site is contaminated with polychlorinated biphenyls (PCBs). PCB oil was utilized in the transformers and in the hydraulic presses. In 1998, EPA contractor Earth Tech, Inc. performed removal of drums, PCB contaminated materials and asbestos from the site. Current site operations by Earth Tech, Inc. involve the demolition of a concrete pad, limited soils excavation and cutting wooden beams to size. RST has been tasked to perform air sampling for PCBs and particulate air monitoring.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The EPA On-Scene Coordinator (OSC), Jack Harmon, will provide overall direction to the staff concerning project sampling needs, objectives, and schedule. The Site Project Manager (SPM), Michael Mahnkopf, will be the primary point of contact with the OSC. The SPM is responsible for the development and completion of the Sampling QA/QC Plan, project team organization, and supervision of all project tasks, including reporting and deliverables. The Site QC Coordinator will be responsible for ensuring field adherence to the Sampling QA/QC Plan and recording of any deviations. The RST Quality Assurance Officer (QAO), Smita Sumbaly, will be the primary project team site contact with the subcontracted laboratory, if necessary.

The ERRS contractor, Earth Tech, Inc. will subcontract to Upstate Laboratories, Inc., E. Syracuse, New York for the performance of all laboratory analysis.

The following personnel will work on this project:

Personnel	**Responsibility**
Jack Harmon	On-Scene Coordinator
Michael Mahnkopf	Site Project Manager, Sample Collection and Management

The following laboratory will provide the following analyses:

Lab Name/Location	Sample Type	Parameters
Upstate Laboratory Inc. 6034 Corporate Drive East Syracuse, NY 13057	Air	PCB

A turnaround time of 24 for the verbal results and two weeks for the written results has been requested by the OSC.

4.0 DATA USE OBJECTIVES, QA OBJECTIVES

In addition to the following, the Data Used Objectives, QA Objectives procedure will be conducted in accordance with Sections A7, B2, B4, and B5 of the Region II RST QAPP.

The objective of this air sampling and monitoring project is to determine if on-site operations are impacting the adjacent residential neighborhood.

4.1 Data Use Objectives

The overall Quality Assurance (QA) objective for chemical measurement data associated with this sampling event is to provide analytical results that are legally defensible in a court of law. The QA program will incorporate Quality Control (QC) procedures for field sampling, chain of custody, laboratory analyses and reporting to assure generation of sound analytical results.

The EPA On-Scene Coordinator (OSC) has specified a level of QA-1 for this sampling event. Details of this QA level are provided below.

4.2 QA Objectives

The QA Protocols for a Level 1 QA objective sampling event are applicable to all sample matrices and include:

1. Sample documentation in the form of field logbooks, appropriate field data sheets, and chain of custody records (chain of custody records are optional for field screening locations).

2. Calibration of all monitoring and/or field-portable analytical equipment prior to collection and analyses of samples with results and/or performance check procedures/methods summarized and documented in a field, personal, and/or instrument log notebook.

3. Field or laboratory determined method detection limits (MDLs) will be recorded along with corresponding analytical sample results, where appropriate.

The objective of this project/event applies to the following parameters:

Table 1: QUALITY ASSURANCE OBJECTIVES

QA Parameters	Matrix	Intended Use of Data	QA Objective
PCB	Air	Determine if on-site operations are impacting the adjacent residential neighborhood	QA-1

A Field Sampling Summary is attached in Table 2 and a QA/QC Analysis and Objectives Summary is attached in Table 3. Section 5.1, Sampling Design, provides information on the analyses to be performed.

TABLE 2:

FIELD SAMPLING SUMMARY

Analytical Parameters	Matrix	Container Size	Preservative	Holding Time[1]	Subtotal Samples	Lot Blanks[2]	Field Blanks[3]	Duplicate Samples	Total Field Samples
PCB	Air	76 mm polyurethane foam (PUF) sorbent	cartridge should be wrapped in aluminum foil to prevent light	Not applicable	15	1	5	1	22

1 Holding time from date of sampling.
2 Not required for QA-1 (screening) and high-concentration liquids.
3 Only required if non-dedicated sampling equipment to be used. NR - not required, dedicated sampling equipment to be used.

TABLE 3

QA/QC ANALYSIS AND OBJECTIVES SUMMARY

Analytical Parameters	Matrix	Analytical Method Reference	QA/QC Quantitation Limits	QA Objective
PCB	Air	EPA Method TO-10A	As per method	QA-1

Note: CLP-format deliverables required for all data packages.

5.0 APPROACH AND SAMPLING PROCEDURES

In addition to the following, the approach and sampling procedures will be conducted in accordance with Sections B1 and B4 of the EPA Region II RST QAPP.

RST will conduct air sampling and particulate air monitoring at the Bossert Manufacturing Site.

This sampling design is based on information currently available and may be modified on site in light of field screening results and other acquired information. All deviations from the sampling plan will be noted in the Sampling Trip Report.

5.1 Sampling Design

RST will conduct outdoor air sampling at three specific perimeter locations during each day of site operations. These sampling locations will be established along the property fence lines in the following way: one location along Noyles Street, one location along Lenox Avenue facing Maple Street and one location along Lenox Avenue facing the Oak Street. An approximate flow rate of 2.0 liters/minute for a 480 minute time period is targeted for this project. The samples will be submitted for PCB analysis via EPA Method TO-10A (see Attachment A).

QA/QC samples will include the submission of one field blank per day. Duplicate samples and lot blank samples will be collected at the rate of one per twenty. Field duplicate samples provide an indication of analytical variability and analytical error and will not be identified to the laboratory.

In addition to air sampling at the above stated locations, total particulate air monitoring will also be performed at each location during each day of site operations.

5.2 Schedule of Activities

Proposed Start Date	Activity	End Date
May 5,2003	Air Sampling and Monitoring	May 9, 2003

5.3 Sampling Equipment

Air samples will be collected utilizing SKC Model 224-PCXR8 sample pumps and 76 mm polyurethane foam (PUF) sorbent tubes. Pump flow rates will be measured (calibrated) before and after sample collection utilizing a BIOS Dry Cell flow meter.

Particulate air monitoring will be performed utilizing MIE DR-2000 Data Rams. Prior to use each day, they will be calibrated in accordance with the manufacturer's instructions.

5.4 Sample Identification System

Each sample collected by Region II RST will be designated by a code which will identify the site. The code will be a site-specific project tracking number. The code for the Bossert Manufacturing Site is *BM*. The site code will be followed by three first letters of the street name where a sample was collected and the sequential sample number. For example, the first sample collected along the Lenox Avenue fence line facing Maple Street will be identified as *BM-LENMAP-1*. The second sample collected along the Lenox Avenue fence line facing Oak Street will be identified as *BM-LENOAK-2*.

A duplicate sample will be identified in the same manner as other samples and will be distinguished and documented in the field logbook.

5.5 Standard Operating Procedures (SOPs)

5.5.1 Sample Documentation

All sample documents will be completed legibly, in ink. Any corrections or revisions will be made by lining through the incorrect entry and by initialing the error.

FIELD LOGBOOK

The field logbook is essentially a descriptive notebook detailing site activities and observations so that an accurate account of field procedures can be reconstructed in the writer's absence. All entries will be dated and signed by the individuals making the entries, and should include (at a minimum) the following:

1. Site name and project number
2. Name(s) of personnel on site
3. Dates and times of all entries (military time preferred)
4. Descriptions of all site activities, site entry and exit times
5. Noteworthy events and discussions
6. Weather conditions
7. Site observations
8. Sample and sample location identification and description*
9. Subcontractor information and names of on-site personnel
10. Date and time of sample collections, along with chain of custody information
11. Record of photographs
12. Site sketches

* - The description of the sample location will be noted in such a manner as to allow the reader to reproduce the location in the field at a later date.

SAMPLE LABELS

Sample labels will clearly identify the particular sample, and should include the following:

1. Site/project number.
2. Sample identification number.
3. Sample collection date and time.
4. Designation of sample (grab or composite).
5. Sample preservation.
6. Analytical parameters.
7. Name of sampler.

Sample labels will be written in indelible ink and securely affixed to the sample container. Tie-on labels can be used if properly secured.

CUSTODY SEALS

Custody seals demonstrate that a sample container has not been tampered with, or opened. The individual in possession of the sample(s) will sign and date the seal, affixing it in such a manner that the container cannot be opened without breaking the seal. The name of this individual, along with a description of the sample packaging, will be noted in the field logbook.

5.5.2 Sampling SOPs

The following Sampling SOP (Attachment B) will be used for this project:

EPA/ERT SOP No. 2008 - General Air Sampling

5.5.3 Sample Handling and Shipment

Each sample will be capped and packaged according to the following protocol. All capped samples will be placed in zip-lock bags and labeled with the sample number, time and date of collection, analyses requested and preservative used. Sealed bags will be placed in the appropriate shipping container and delivered to the lab. All packaging will conform to IATA Transportation regulations for overnight carriers.

All sample documents will be sealed in a plastic bag and affixed to the underside of each shipping container. The lid will be sealed and affixed on at least two sides with custody seals so that any sign of tampering is easily visible.

5.6 Sample Containers

All sample containers will meet the QA/QC specifications in OSWER Directive 9240.0-05A, "Specifications and Guidance for Contaminant Free Sample Containers".

5.7 Disposal of PPE and Contaminated Sampling Materials

It is not anticipated that any used PPE and/or disposable sampling equipment will be generated during this sampling event.

6.0 SAMPLE CUSTODY

In addition to the following, the Sample Custody procedure will be conducted in accordance with Section B3 of the Region II RST QAPP.

A chain of custody record will be maintained from the time the sample is taken to its final deposition. Every transfer of custody must be noted and signed for, and a copy of this record kept by each individual who has signed. When samples (or groups of samples) are not under direct control of the individual responsible for them, they must be stored in a locked container sealed with a custody seal. Specific information regarding custody of the samples projected to be collected on the weekend will be noted in the field logbook.

The chain of custody record should include (at minimum) the following:

1. Sample identification number
2. Sample information
3. Sample location
4. Sample date
5. Name(s) and signature(s) of sampler(s)
6. Signature(s) of any individual(s) with custody of samples

A separate chain of custody form must accompany each shipping container for each daily shipment. The chain of custody form must address all samples in that cooler, but not address samples in any other shipping container. This practice maintains the chain of custody for all samples in case of mis-shipment.

7.0 FIELD INSTRUMENT CALIBRATION AND PREVENTIVE MAINTENANCE

In addition to the following, the Field Instrument and Preventative Maintenance procedure will be conducted in accordance with Section B6 of the Region II RST QAPP.

The sampling team is responsible for assuring that a calibration/maintenance log will be brought into the field and maintained for each measuring device. Each log will include at a minimum, where applicable:

- name of device and/or instrument calibrated
- device/instrument serial and/or ID number
- frequency of calibration
- date of calibration
- results of calibration
- name of person performing the calibration
- identification of the calibrant

Equipment to be used each day will be calibrated prior to the commencement of daily activities.

8.0 ANALYTICAL METHODS

Analytical methods to be utilized in the analyses of samples collected during this sampling event are detailed in Table 3.

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

In addition to the following, the Data Reduction, Validation, and Reporting procedure will be conducted in accordance with Sections D1, D2, and D3 of the Region II RST QAPP.

9.1 Deliverables

The RST SPM, Michael Mahnkopf, will maintain contact with the EPA OSC, Jack Harmon to keep him informed about the technical and financial progress of this project. This communication will commence with the issuance of the work assignment and project scoping meeting. Activities under this project will be reported in status and trip reports and other deliverables (e.g., analytical reports, final reports) described herein. Activities will also be summarized in appropriate format for inclusion in monthly and annual reports.

The following deliverables will be provided under this project:

TRIP REPORT

A trip report will be prepared to provide a detailed accounting of what occurred during each sampling mobilization. The trip report will be prepared within one week of the last day of each sampling mobilization. Information will be provided on time of major events, dates, and personnel on site (including affiliations).

MAPS/FIGURES

Maps depicting site layout, contaminant source areas, and sample locations will be included in the trip report, as appropriate.

ANALYTICAL REPORT

An analytical report will be prepared by Upstate Laboratories, Inc. for samples analyzed under this plan. Information regarding the analytical methods or procedures employed, sample results, QA/QC results, chain of custody documentation, laboratory correspondence, and raw data will be provided within this deliverable.

DATA REVIEW

A review of the data generated under this plan will be undertaken. The assessment of data acceptability or usability will be provided separately, or as part of the analytical report.

9.2 Data Validation

Due to the quality assurance level (QA-1) associated with this project, data validation is not required.

10.0 FIELD QUALITY CONTROL CHECKS AND FREQUENCY

In addition to the following, the Field Quality Control Checks and Frequency procedure will be conducted in accordance with Section B7 of the Region II RST QAPP.

This section details the Quality Assurance/Quality Control (QA/QC) requirements for field activities performed during the sampling effort.

QA/QC samples will include the collection of one field duplicate and one lot blank at a frequency of 1 per 20 samples. Field duplicate samples provide an indication of analytical variability and analytical error and will not be identified to the laboratory. Field blanks will be submitted at the frequency of one per day. Field blanks place a mechanism of control on sample handling, storage and shipment. Field blanks are also indicative of ambient conditions and/or equipment conditions that may potentially affect the quality of the associated samples.

11.0 SYSTEM AUDIT

In addition to the following, the System Audit procedure will be conducted in accordance with Section C1 of the Region II RST QAPP.

The Field QA/QC Officer will observe sampling operations and review subsequent analytical results to ensure compliance with the QA/QC requirements of the project/sampling event.

12.0 CORRECTIVE ACTION

In addition to the following, the Corrective Action procedure will be conducted in accordance with Section C1 of the Region II RST QAPP.

All provisions will be taken in the field and laboratory to ensure that any problems that may develop will be dealt with as quickly as possible to ensure the continuity of the project/sampling events. Any deviations from this sampling plan will be noted in the final report.

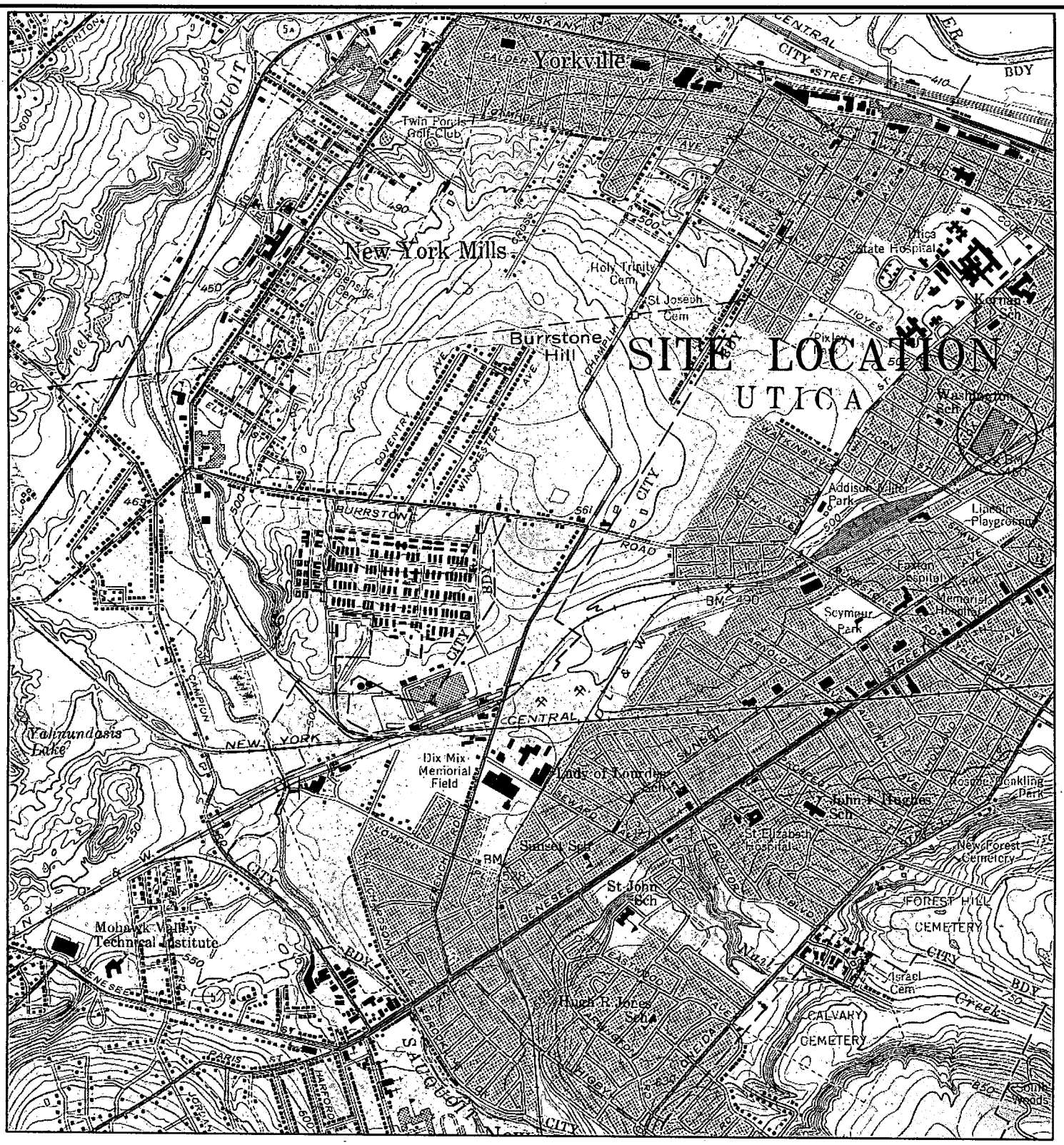

FIGURE 1
SITE LOCATION MAP
BOSSERT MANUFACTURING
UTICA, NY

US ENVIRONMENTAL PROTECTION AGENCY
REMOVAL SUPPORT TEAM
CONTRACT # 68-W-00-113

EDITED BY: W. HENSPERGER	
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ATTACHMENT A

EPA Method TO-10A

EPA/625/R-96/010b

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

Second Edition

Compendium Method TO-10A

Determination Of Pesticides And Polychlorinated Biphenyls In Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD)

Center for Environmental Research Information
Office of Research and Development
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Cincinnati, OH 45268

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Method TO-10A
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- John Burckle, U.S. EPA, ORD, Cincinnati, OH
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- Robert G. Lewis, U.S. EPA, NERL, RTP, NC
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- Frank F. McElroy, U.S. EPA, NERL, RTP, NC
- Heidi Schultz, ERG, Lexington, MA
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

Method TO-10 was originally published in March of 1989 as one of a series of peer reviewed methods in the second supplement to *"Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air,"* EPA 600/4-89-018. In an effort to keep these methods consistent with current technology, Method TO-10 has been revised and updated as Method TO-10A in this Compendium to incorporate new or improved sampling and analytical technologies. In addition, this method incorporates ASTM Method D 4861-94, *Standard Practice for Sampling and Analysis of Pesticides and Polychlorinated Biphenyls in Air.*

This Method is the result of the efforts of many individuals. Gratitude goes to each person involved in the preparation and review of this methodology.

Author(s)

- Robert G. Lewis, U.S. EPA, NERL, RTP, NC

Peer Reviewers

- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC
- Irene D. DeGraff, Supelco, Bellefonte, PA
- Lauren Drees, U.S. EPA, NRMRL, Cincinnati, OH

Finally, recognition is given to Frances Beyer, Lynn Kaufman, Debbie Bond, Cathy Whitaker, and Kathy Johnson of Midwest Research Institute's Administrative Services staff whose dedication and persistence during the development of this manuscript has enabled it's production.

DISCLAIMER

METHOD TO-10A

Determination Of Pesticides And Polychlorinated Biphenyls In Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD)

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METHOD TO-10A

Determination Of Pesticides And Polychlorinated Biphenyls In Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector (GC/MD) Detection

1. Scope

1.1 This document describes a method for sampling and analysis of a variety of common pesticides and for polychlorinated biphenyls (PCBs) in ambient air. The procedure is based on the adsorption of chemicals from ambient air on polyurethane foam (PUF) or a combination of PUF and granular sorbent using a low volume sampler.

1.2 The low volume PUF sampling procedure is applicable to multicomponent atmospheres containing common pesticide concentrations from 0.001 to 50 $\mu g/m^3$ over 4- to 24-hour sampling periods. The limits of detection will depend on the nature of the analyte and the length of the sampling period.

1.3 Specific compounds for which the method has been employed are listed in Table 1. The analytical methodology described in Compendium Method TO-10A is currently employed by laboratories throughout the U.S. The sampling methodology has been formulated to meet the needs of common pesticide and PCB sampling in ambient air.

1.4 Compendium Method TO-10 was originally published in 1989. The method was further modified for indoor air application in 1990. In an effort to keep the method consistent with current technology, Compendium Method TO-10 has incorporated ASTM Method D4861-94 (1) and is published here as Compendium Method TO-10A.

2. Summary of Method

2.1 A low-volume (1 to 5 L/minute) sample is used to collect vapors on a sorbent cartridge containing PUF or PUF in combination with another solid sorbent. Airborne particles may also be collected, but the sampling efficiency is not known (2).

2.2 Pesticides and other chemicals are extracted from the sorbent cartridge with 5 percent diethyl ether in hexane and determined by gas chromatography coupled with an electron capture detector (ECD), nitrogen-phosphorus detector (NPD), flame photometric detector (FPD), Hall electrolytic conductivity detector (HECD), or a mass spectrometer (MS). For common pesticides, high performance liquid chromatography (HPLC) coupled with an ultraviolet (UV) detector or electrochemical detector may be preferable. This method describes the use of an electron capture detector.

2.3 Interferences resulting from analytes having similar retention times during GC analysis are resolved by improving the resolution or separation, such as by changing the chromatographic column or operating parameters, or by fractionating the sample by column chromatography.

3. Significance

3.1 Pesticide usage and environmental distribution are common to rural and urban areas of the United States. The application of pesticides can cause potential adverse health effects to humans by contaminating soil, water, air, plants, and animal life. However, human exposure to PCBs continues to be a problem because of their presence in the environment.

3.2 Many pesticides and PCBs exhibit bioaccumulative, chronic health effects; therefore, monitoring the presence of these compounds in ambient air is of great importance.

3.3 Use of a portable, low volume PUF sampling system allows the user flexibility in locating the apparatus. The user can place the apparatus in a stationary or mobile location. The portable sampling apparatus may be positioned in a vertical or horizontal stationary location (if necessary, accompanied with supporting structure). Mobile positioning of the system can be accomplished by attaching the apparatus to a person to test air in the individual's breathing zone.

3.4 Moreover, this method has been successfully applied to measurement of common pesticides in outdoor air, indoor air and for personal respiratory exposure monitoring (3).

4. Applicable Documents

4.1 ASTM Standards

- D1356 *Definition of Terms Relating to Atmospheric Sampling and Analysis*
- D4861-94 *Standard Practice for Sampling and Analysis of Pesticides and Polychlorinated Biphenyls in Air*
- E260 *Recommended Practice for General Gas Chromatography Procedures*
- E355 *Practice for Gas Chromatography Terms and Relationships*
- D3686 *Practice for Sampling Atmospheres to Collect Organic Compound Vapors (Activated Charcoal Tube Adsorption Method*
- D3687 *Practice for Analysis of Organic Compound Vapors Collected by the Activated Charcoal Tube Adsorption*
- D4185 *Practice for Measurement of Metals in Workplace Atmosphere by Atomic Absorption Spectrophotometry*

4.2 EPA Documents

- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air: Method TO-10, Second Supplement,* U. S. Environmental Protection Agency, EPA 600/4-89-018, March 1989.
- *Manual of Analytical Methods for Determination of Pesticides in Humans and Environmental Standards*, U. S. Environmental Protection Agency, EPA 600/8-80-038, June 1980.
- *Compendium of Methods for the Determination of Air Pollutants in Indoor Air: Method IP-8,* U. S. Environmental Protection Agency, EPA 600/4-90-010, May 1990.

4.3 Other Documents

- Code of Federal Regulations, Title 40, Part 136, Method 604

5. Definitions

[Note: Definitions used in this document and in any user-prepared Standard operating procedures (SOPs) should be consistent with ASTM D1356, E260, and E355. All abbreviations and symbols are defined within this document at point of use.]

5.1 Sampling efficiency (SE)-ability of the sampling medium to trap analytes of interest. The percentage of the analyte of interest collected and retained by the sampling medium when it is introduced as a vapor in air or nitrogen into the air sampler and the sampler is operated under normal conditions for a period of time equal to or greater than that required for the intended use is indicated by %SE.

5.2 Retention efficiency (RE)-ability of sampling medium to retain a compound added (spiked) to it in liquid solution.

5.3 Static retention efficiency-ability of the sampling medium to retain the solution spike when the sample cartridge is stored under clean, quiescent conditions for the duration of the test period.

5.4 Dynamic retention efficiency (RE_d)-ability of the sampling medium to retain the solution spike when air or nitrogen is drawn through the sampling cartridge under normal operating conditions for the duration of the test period. The dynamic RE is normally equal to or less than the SE.

5.5 Retention time (RT)-time to elute a specific chemical from a chromatographic column, for a specific carrier gas flow rate, measured from the time the chemical is injected into the gas stream until it appears at the detector.

5.6 Relative retention time (RRT)-a rate of RTs for two chemicals for the same chromatographic column and carrier gas flow rate, where the denominator represents a reference chemical.

5.7 Surrogate standard-a chemically inert compound (not expected to occur in the environmental sample) that is added to each sample, blank, and matrix-spiked sample before extraction and analysis. The recovery of the surrogate standard is used to monitor unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within acceptable limits.

6. Interferences

6.1 Any gas or liquid chromatographic separation of complex mixtures of organic chemicals is subject to serious interference problems due to coelution of two or more compounds. The use of capillary or microbore columns with superior resolution or two or more columns of different polarity will frequently eliminate these problems. In addition, selectivity may be further enhanced by use of a MS operated in the selected ion monitoring (SIM) mode as the GC detector. In this mode, co-eluting compounds can often be determined.

6.2 The ECD responds to a wide variety of organic compounds. It is likely that such compounds will be encountered as interferences during GC/ECD analysis. The NPD, FPD, and HECD detectors are element specific, but are still subject to interferences. UV detectors for HPLC are nearly universal, and the electrochemical detector may also respond to a variety of chemicals. Mass spectrometric analyses will generally provide positive identification of specific compounds.

6.3 PCBs and certain organochlorine pesticides (e.g., chlordane) are complex mixtures of individual compounds which can cause difficulty in accurately quantifying a particular formulation in a multiple component mixture. PCBs may interfere with the determination of pesticides.

6.4 Contamination of glassware and sampling apparatus with traces of pesticides or PCBs can be a major source of error, particularly at lower analyte concentrations. Careful attention to cleaning and handling procedures is required during all steps of sampling and analysis to minimize this source of error.

6.5 The general approaches listed below should be followed to minimize interferences.

6.5.1 Polar compounds, including certain pesticides (e.g., organophosphorus and carbamate classes) can be removed by column chromatography on alumina. Alumina clean-up will permit analysis of most organochlorine pesticides and PCBs (4).

6.5.2 PCBs may be separated from other organochlorine pesticides by column chromatography on silicic acid (5,6).

6.5.3 Many pesticides can be fractionated into groups by column chromatography on Florisil (6).

7. Equipment and Materials

7.1 Materials for Sample Collection

7.1.1 Continuous-Flow Sampling Pump (see Figure 1). The pump should provide a constant air flow ($\leq \pm 5\%$), be quiet and unobtrusive, with a flow rate of 1 to 5 L/min. Sources of equipment are Supelco, Supelco Park, Bellefonte, PA; SKC, 334 Valley View Road, Eighty Four, PA and other manufacturers.

7.1.2 Sampling Cartridge (see Figure 2). Constructed from a 20-mm (I.D.) x 10-cm borosilicate glass tube drawn down to a 7-mm (O.D.) open connection for attachment to the pump by way of flexible tubing (see Figure 1).

7.1.3 Sorbent, Polyurethane Foam (PUF). Cut into a cylinder, 22-mm I.D. and 7.6-cm long, fitted under slight compression inside the cartridge. The PUF should be of the polyether type, (density of 0.0225 g/cm^3). This is the type of foam used for furniture upholstery, pillows, and mattresses. The PUF cylinders (plugs) should be slightly larger in diameter than the internal diameter of the cartridge. The PUF sorbent may be cut by one of the following means:

- With a high-speed cutting tool, such as a motorized cork borer. Distilled water should be used to lubricate the cutting tool.
- With a hot wire cutter. Care should be exercised to prevent thermal degradation of the foam.
- With scissors, while plugs are compressed between the 22-mm circular templates.

Alternatively, pre-extracted PUF plugs and glass cartridges may be obtained commercially.

7.1.4 Particle Filter. The collection efficiency of PUF for small-diameter (0.1 to 1 μm) airborne particles is only about 20% (7). However, most pesticides and PCBs exist in air under steady-state conditions primarily as vapors (8). Most particulate-associated pesticides or PCBs, if any, will also tend to be vaporized from filters after collection (9). Collocated sampling with and without a quartz-fiber pre-filter has yielded indistinguishable results for a broad spectrum of pesticides and PCBs found in indoor air (10).

7.1.4.1 An open-face filter may be attached to the sampling cartridge by means of a union for 1-in. (25.4-mm) tubing.

7.1.4.2 A 32-mm diameter quartz microfiber filter (e.g., Palifelex® type 2500 QAT-UP) is placed in the open end of the union and supported by means of a screen or perforated metal plate [e.g., a 304-stainless steel disk, 0.0312-in. (0.8-mm) thick with 1/16-in. (1.6-mm) diameter round perforations at 132 holes per in.^{2} (20 holes/cm^2), 41% open area.]. A 32-mm Viton® O-ring is placed between the filter and outer nut to effect a seal (see Figure 3). This filter holder is available from Supelco Park, Bellefonte, PA; SKC, 334 Forty Eight, PA; and other manufacturers.

7.1.5 Size-Selective Impactor Inlet. A size-selective impactor inlet with an average particle-size cut-point of 2.5 μm or 10 μm mean diameter at a sampling rate of 4 L/min may be used to exclude nonrespirable airborne particulate matter (11). This inlet, particle filter support, sampling cartridge holders are available commercially from Supelco, Supelco Park, Bellefonte, PA; SKC, 334 Forty Eight, PA and University Research Glassware (URG), Chapel Hill, NC.

7.1.6 Tenax-TA. 60/80 mesh, 2,6-diphenylphenylene oxide polymer. Commercially available from Supelco, Supelco Park, Bellefonte, PA and SKC, 334 Forty Eight, PA.

7.2 Equipment for Analysis

7.2.1 Gas Chromatograph (GC). The GC system should be equipped with appropriate detector(s) and either an isothermally controlled or temperature programmed heating oven. Improved detection limits may be obtained with a GC equipped with a cool on-column or splitless injector.

7.2.2 Gas Chromatographic Column. As an example, a 0.32 mm (I.D.) x 30 m DB-5, DB-17, DB-608, and DB-1701 are available. Other columns may also provide acceptable results.

7.2.3 HPLC Column. As an example, a 4.6-mm x 25-cm Zorbax SIL or μBondpak C-18. Other columns may also provide acceptable results.

7.2.4 Microsyringes. 5 μL volume or other appropriate sizes.

7.3 Reagents and Other Materials

7.3.1 Round Bottom Flasks. 500 mL, T 24/40 joints, best source.
7.3.2 Capacity Soxhlet Extractors. 300 mL, with reflux condensers, best source.
7.3.3 Kuderna-Danish Concentrator. 500 mL, with Snyder columns, best source.
7.3.4 Graduated Concentrator Tubes. 10 mL, with 19/22 stoppers, best source.
7.3.5 Graduated Concentrator Tubes. 1 mL, with 14/20 stoppers, best source.
7.3.6 TFE Fluorocarbon Tape. 1/2 in., best source.
7.3.7 Filter Tubes. Size 40 mm (I.D.) x 80 mm.
7.3.8 Serum Vials. 1 mL and 5 mL, fitted with caps lined with TFE fluorocarbon.
7.3.9 Pasteur Pipettes. 9 in., best source.
7.3.10 Glass Wool. Fired at 500°C, best source.
7.3.11 Boiling Chips. Fired at 500°C, best source..
7.3.12 Forceps. Stainless steel, 12 in., best source.
7.3.13 Gloves. Latex or precleaned (5% ether/hexane Soxhlet extracted) cotton.

7.3.14 Steam Bath.
7.3.15 Heating Mantles. 500 mL.
7.3.16 Analytical Evaporator. Nitrogen blow-down.
7.3.17 Acetone. Pesticide quality.
7.3.18 n-Hexane. Pesticide quality.
7.3.19 Diethyl Ether. Preserved with 2% ethanol.
7.3.20 Sodium Sulfate. Anhydrous analytical grade.
7.3.21 Alumina. Activity Grade IV, 100/200 mesh.
7.3.22 Glass Chromatographic Column. 2-mm I.D. x 15-cm long.
7.3.23 Soxhlet Extraction System. Including Soxhlet extractors (500 and 300 mL), variable voltage transformers, and cooling water source.
7.3.24 Vacuum Oven. Connected to water aspirator.
7.3.25 Die.
7.3.26 Ice Chest.
7.3.27 Silicic Acid. Pesticide grade.
7.3.28 Octachloronaphthalene (OCN). Research grade.
7.3.29 Florisil. Pesticide grade.

8. Assembly and Calibration of Sampling System

8.1 Description of Sampling Apparatus

8.1.1 A typical sampling arrangement utilizing a personal air pump is shown in Figure 1. This method is designed to use air sampling pumps capable of pulling air through the sampling cartridge at flow rates of 1 to 5 L/min. The method writeup presents the use of this device.
8.1.2 The sampling cartridge (see Figure 2) consists of a glass sampling cartridge in which the PUF plug or PUF/Tenax® TA "sandwich" is retained.

8.2 Calibration of Sampling System

8.2.1 Air flow through the sampling system is calibrated by the assembly shown in Figure 4. All air sampler must be calibrated in the laboratory before and after each sample collection period, using the procedure described below.
8.2.2 For accurate calibration, attach the sampling cartridge in-line during calibration. Vinyl bubble tubing or other means (e.g., rubber stopper or glass joint) may be used to connect the large end of the cartridge to the calibration system. Refer to ASTM Practice D3686 or D4185, for procedures to calibrate small volume air pumps.

9. Preparation of PUF Sampling Cartridges

9.1 The PUF adsorbent is white and yellows upon exposure to light. The "yellowing" of PUF will not affect its ability to collected pesticides or PCBs.
9.2 For initial cleanup and quality assurance purposes, the PUF plug is placed in a Soxhlet extractor and extracted with acetone for 14 to 24 hours at 4 to 6 cycles per hour.

[Note: If commercially pre-extracted PUF plugs are used, extraction with acetone is not required.]

Follow with a 16-hour Soxhlet extraction with 5% diethyl ether in n-hexane. When cartridges are reused, 5% diethyl ether in n-hexane can be used as the cleanup solvent.

9.3 Place the extracted PUF in a vacuum oven connected to a water aspirator and dry at room temperature for 2 to 4 hours (until no solvent odor is detected). Alternatively, they may be dried at room temperature in an air-tight container with circulating nitrogen (zero grade). Place the clean PUF plug into a labeled glass sampling cartridges using gloves and forceps. Wrap the cartridges with hexane-rinsed aluminum foil and placed in jars fitted with TFE fluorocarbon-lined caps. The foil wrapping may also be marked for identification using a blunt probe.

9.4 Granular sorbents may be combined with PUF to extend the range of use to compounds with saturation vapor pressures greater than 10^{-4} kPa (6). A useful combination trap can be assembled by "sandwiching" 0.6 g of Tenax-TA between two 22-mm I.D. x 3.8-cm pre-cleaned PUF plugs, as shown in Figure 2, Cartridge b. The Tenax-TA should be pre-extracted as described in Section 9.2. This trap may be extracted, vacuum dried, and removed without unloading it.

9.5 Analyze at least one assembled cartridge from each batch as a laboratory blank before the batch is acceptable. A blank level of <10 ng/plug for single component compounds is considered to be acceptable. For multiple component mixtures (e.g., PCBs), the blank level should be <100 ng/plug.

9.6 After cleaning, cartridges are considered clean up to 30 days when stored in sealed containers. Certified clean cartridges do not need to be chilled when shipping to the field.

10. Sampling

[Note: After the sampling system has been assembled and calibrated as per Section 8, it can be used to collect air samples as described below. The prepared sample cartridges should be used within 30 days of certification and should be handled only with latex or precleaned cotton gloves.]

10.1 Carefully remove the clean sample cartridge from the aluminum foil wrapping (the foil is returned to jars for later use) and attached to the pump with flexible tubing. The sampling assembly is positioned with the intake downward or in horizontal position. Locate the sampler in an unobstructed area at least 30 meters from any obstacle to air flow. The PUF or PUF/XAD-2 cartridge intake is positioned 1 to 2 m above ground level. Cartridge height above ground is recorded on the Compendium Method TO-10A field test data sheet (FTDS), as illustrated in Figure 5.

10.2 After the PUF cartridge is correctly inserted and positioned, the power switch is turned on and the sampling begins. The elapsed time meter is activated and the start time is recorded. The pumps are checked during the sampling process and any abnormal conditions discovered are recorded on the FTDS. Ambient temperatures and barometric pressures are measured and recorded periodically during the sampling procedure on the FTDS.

10.3 At the end of the desired sampling period, the power is turned off, the PUF cartridge removed from the sampler and wrapped with the original aluminum foil and placed in a sealed, labeled container for transport, under blue ice (<4°C), back to the laboratory. At least one field blank is returned to the laboratory with each group of

samples. A field blank is treated exactly like a sample except that no air is drawn through the cartridge. Samples are stored at <4°C or below until analyzed in the laboratory. Extraction must occur within 7 days of sampling and analysis within 40 days of extraction. Refer to ASTM D4861-94 (1), Appendix X3 for storage stability for various common pesticides and other compounds on PUF or PUF/Tenax TA sandwich.

11. Sample Extraction Procedure

[Note: Sample extraction should be performed under a properly ventilated hood.]

11.1 Sample Extraction

11.1.1 All samples should be extracted within 1 week after collection. All samples should be stored at <4°C until extracted.

11.1.2 All glassware should be washed with a suitable detergent; rinsed with deionized water, acetone, and hexane; rinsed again with deionized water; and fired in an oven (500°C).

11.1.3 Prepare a spiking solution for determination of extraction efficiency. The spiking solution should contain one or more surrogate compounds that have chemical structures and properties similar to those of the analytes of interest. Octachloronaphthalene (OCN) and dibutylchlorendate have been used as surrogates for determination of organochlorine pesticides by GC with an ECD. Tetrachloro-m-xylene and decachlorobiphenyl can also be used together to insure recovery of early and late eluting compounds. For organophosphate pesticides, tributylphosphate or triphenylphosphate may be employed as surrogates. The surrogate solution should be prepared so that addition of 100 μL into the PUF plug results in an extract containing the surrogate compound at the high end of the instrument's calibration range. As an example, the spiking solution for OCN is prepared by dissolving 10 mg of OCN in 10 mL of 10% acetone in n-hexane, followed by serial dilution n-hexane to achieve a final spiking solution of OCN of 1 μg/mL.

[Note: Use the recoveries of the surrogate compounds to monitor for unusual matrix effects and gross sample processing errors. Evaluate surrogate recovery for acceptance by determining whether the measured concentration falls within the acceptance limits of 60-120 percent.]

11.1.4 The extracting solution (5% diethyl ether/hexane) is prepared by mixing 1900 mL of freshly opened hexane and 100 mL of freshly opened diethyl ether (preserved with ethanol) to a flask.

11.1.5 All clean glassware, forceps, and other equipment to be used should be rinsed with 5% diethyl ether/hexane and placed on rinsed (5% diethyl ether/hexane) aluminum foil until use. The condensing towers should also be rinsed with 5% diethyl ether/hexane. Then add 300 mL or 5% diethyl ether/hexane to the 500 mL round bottom boiling flask and add up to three boiling granules.

11.1.6 Using precleaned (i.e., 5% diethyl ether/hexane Soxhlet extracted) cotton gloves, the glass PUF cartridges are removed from the sealed container, the PUF removed from the glass container and is placed into the 300 mL Soxhlet extractor using prerinsed forceps.

[Note: If "sandwich" trap is used, carefully clean outside walls of cartridge with hexane-soaked cotton swabs or laboratory tissues (discard) and place cartridge into extractor with intake (large end) downward.]

11.1.7 Before extraction begins, add 100 µL of the OCN solution directly to the top of the PUF plug.

[Note: Incorporating a known concentration of the solution onto the sample provides a quality assurance check to determine recovery efficiency of the extraction and analytical processes.]

11.1.8 Connect the Soxhlet extractor to the 500 mL boiling flask and condenser. Wet the glass joints with 5% diethyl ether/hexane to ensure a tight seal between the fittings. If necessary, the PUF plug can be adjusted using forceps to wedge it midway along the length of the siphon. The above procedure should be followed for all samples, with the inclusion of a blank control sample.

11.1.9 The water flow to the condenser towers of the Soxhlet extraction assembly should be checked and the heating unit turned on. As the samples boil, the Soxhlet extractors should be inspected to ensure that they are filling and siphoning properly (4 to 6 cycles/hour). Samples should cycle for a minimum of 16 hours.

11.1.10 At the end of the extracting process (minimum of 16 hours), the heating unit is turned off and the sample cooled to room temperature.

11.1.11 The extracts are then concentrated to 5 mL using a Kuderna-Danish (K-D) apparatus. The K-D is set up, assembled with concentrator tubes, and rinsed. The lower end of the filter tube is packed with glass wool and filled with sodium sulfate to a depth of 40 mm. The filter tube is then placed in the neck of the K-D. The Soxhlet extractors and boiling flasks are carefully removed from the condenser towers and the remaining solvent is drained into each boiling flask. Sample extract is carefully poured through the filter tube into the K-D. Each boiling flask is rinsed three times by swirling hexane along the sides. Once the sample has drained, the filter tube is rinsed down with hexane. Each Synder column is attached to the K-D and rinsed to wet the joint for a tight seal. The complete K-D apparatus is placed on a steam bath and the sample is evaporated to approximately 5 mL.

[Note: Do not allow samples to evaporate to dryness.]

Remove sample from the steam bath, rinse Synder column with minimum of hexane, and allow to cool. Adjust sample volume to 10 mL in a concentrator tube, close with glass stopper and seal with TFE fluorocarbon tape. Alternatively, the sample may be quantitatively transferred (with concentrator tube rinsing) to prescored vials and brought up to final volume. Concentrated extracts are stored at <4 °C until analyzed. Analysis should occur no later than 40 days after sample extraction.

11.2 Sample Cleanup

11.2.1 If polar compounds (from example, organophosphorus and carbamate classes) that interfere with GC/ECD analysis are present, use column chromatographic cleanup or alumina. The sample cleanup will permit the analysis of most organochlorine pesticides or PCBs.

11.2.2 Before cleanup, the sample extract is carefully reduced to 1 mL using a gentle stream of clean nitrogen.

11.2.3 A glass chromatographic column (2-mm I.D. x 15-cm long) is packed with alumina, activity grade IV, and rinsed with approximately 20 mL of n-hexane. The concentrated sample extract is placed on the column and eluted with 10 mL of n-hexane at a rate of 0.5 mL/minute. The eluate volume is adjusted to exactly 10 mL and analyzed as per Section 12.

11.2.4 If both PCBs and organochlorine pesticides are sought, alternate cleanup procedures (5,6) may be required (i.e., silicic acid).

11.2.5 Finally, class separation and improved specificity can be achieved by column clean-up and separation on Florisil (6).

12. Analytical Procedure

12.1 Analysis of Organochlorine Pesticides by Capillary Gas Chromatography with Electron Capture Detector (GC/ECD)

[Note: Organochlorine pesticides, PCBs and many nonchlorinated pesticides are responsive to electron capture detection (see Table 1). Most of these compounds can be analyzed at concentration of 1 to 50 ng/mL by GC/ECD. The following procedure is appropriate. Analytical methods that have been used to determine pesticides and PCBs collected from air by this procedure have been published (12).]

12.1.1 Select GC column (e.g., 0.3-mm by 30-m DB-5 column) and appropriate GC conditions to separate the target analytes. Typical operating parameters for this column with splitless injection are: Carrier gas-chromatography grade helium at a flow rate of 1 to 2 mL/min and a column head pressure of 7 to 9 psi (48 to 60 kPa); injector temperature of 250°C; detector temperature of 350°C; initial oven temperature of 50°C held for 2.0 min., ramped at 15°C/min to 150°C for 8 min, ramped at 10°C/min to 295°C then held for 5 min; purge time of 1.0 min. A typical injection volume is 2 to 3 μL.

12.1.2 Remove sample extract from the refrigerator and allow to warm to room temperature.

12.1.3 Prepare standard solution from reference materials of known purity. Analytically pure standards of organochlorine pesticides and PCBs are available from several commercial sources.

12.1.4 Use the standard solutions of the various compounds of interest to determine relative retention times (RRTs) to an internal standard such as p,p'-DDE, aldrin or octachloronaphthalene. Use 1 to 3-μL injections or other appropriate volumes.

12.1.5 Determine detector linearity by injecting standard solutions of three different concentrations (amounts) that bracket the range of analyses. The calibration is considered linear if the relative standard deviation (RSD) of the response factors for the three standards is 20 percent or less.

12.1.6 Calibrate the system with a minimum of three levels of calibration standards in the linear range. The low standard should be near the analytical method detection limit. The calibration is considered linear if the relative standard deviation (RSD) of the response factors for the three standards is 20 percent or less. The initial calibration should be verified by the analysis of a standard from an independent source. Recovery of 85 to 115 percent is acceptable. The initial calibration curve should be verified at the begining of each day and after every ten samples by the analysis of the mid point standard; an RPD of 15% or less is acceptable for continuing use of the initial calibration curve.

12.1.7 Inject 1 to 3 μL of the sample extract. Record volume injected to the nearest 0.05 μL.

12.1.8 A typical ECD response for a mixture of single component pesticides using a capillary column is illustrated in Figure 6. If the response (peak height or area) exceeds the calibration range, dilute the extract and reanalyze.

12.1.9 Quantify PCB mixtures by comparison of the total heights or areas of GC peaks (minimum of 5) with the corresponding peaks in the best-matching standard. Use Aroclor 1242 for early-eluting PCBs and either Aroclor 1254 or Aroclor 1260 as appropriate for late-eluting PCBs.

12.1.10 If both PCBs and organochlorine pesticides are present in the same sample, use column chromatographic separation on silicic acid (5,6) prior to GC analysis.

12.1.11 If polar compounds are present that interfere with GC/ECD analysis, use column chromatographic cleanup or alumina, activity grade IV, in accordance with Section 11.2.

12.1.12 For confirmation use a second GC column such as DB-608. All GC procedures except GC/MS require second column confirmation.

12.1.13 For improved resolution use a capillary column such as an 0.25-mm I.D. x 30-m DB-5 with 0.25 μm film thickness. The following conditions are appropriate.

- Helium carrier gas at 1 mL/min.
- Column temperature program, 90°C (4 min)/16°C/min to 154°C/4°C/min to 270°C.
- Detector, ^{63}Ni ECD at 350°C.
- Make up gas, nitrogen, or 5% methane/95% argon at 60 mL/min.
- Splitless injection, 2 μL maximum.
- Injector temperature, 220°C.

12.1.14 Class separation and improved specificity can be achieved by column chromatographic separation on Florisil (6).

12.2 Analysis of Organophosphorus Pesticides by Capillary Gas Chromatography with Flame Photometric or Nitrogen-Phosphorus Detectors (GC/FPD/NPD)

[Note: Organophosphorus pesticides are responsive to flame photometric and nitrogen-phosphorus (alkali flame ionization) detection. Most of these compounds can be analyzed at concentrations of 50 to 500 ng/mL using either of these detectors.]

12.2.1 Procedures given in Section 12.1.1 through 12.1.9 and Section 12.1.13 through 12.1.14 apply, except for the selection of surrogates.

12.2.2 Use tributylphosphate, triphenylphosphate, or other suitable compound(s) as surrogates to verify extraction efficiency and to determine RRTs.

12.3 Analysis of Carbamate and Urea Pesticides by Capillary Gas Chromatography with Nitrogen-Phosphorus Detector

12.3.1 Trazine, carbamate, and urea pesticides may be determined by capillary GC (DB-5, DB-17, or DB-1701 stationary phase) using nitrogen-phosphorus detection or MS-SIM with detection limits in the 0.05 to 0.2 μL/mL range. Procedures given in Section 12.1.1 through 12.1.9 and Section 12.1.13 through 12.1.14 apply, except for the selection of surrogates, detector, and make up gas.

12.3.2 Thermal degradation may be minimized by reducing the injector temperature to 200°C. HPLC may also be used, but detection limits will be higher (1 to 5 μg/mL).

12.3.3 N-methyl carbamates may be determined using reverse-phase high performance liquid chromatography (HPLC) (C-18) (Section 12.4) and post-column derivatization with o-phthaldehyde and fluorescence detection (EPA Method 531). Detection limits of 0.01 to 0.1 μg/mL can be achieved.

12.4 Analysis of Carbamate, Urea, Pyrethroid, and Phenolic Pesticides by High Performance Liquid Chromatography (HPLC)

[Note: Many carbamate pesticides, urea pesticides, pyrethrins, phenols, and other polar pesticides may be analyzed by high HPLC with fixed or variable wavelength UV detection. Either reversed-phase or normal phase chromatography may be used. Detection limits are 0.2 to 10 μg/mL of extract.]

12.4.1 Select HPLC column (i.e., Zorbax-SIL, 46-mm I.D. x 25-cm, or μ-Bondapak C18, 3.9-mm x 30-cm, or equivalent).

12.4.2 Select solvent system (i.e., mixtures of methanol or acetonitrile with water or mixtures of heptane or hexane with isopropanol).

12.4.3 Follow analytical procedures given in Sections 12.1.2 through 12.1.9.

12.4.4 If interferences are present, adjust the HPLC solvent system composition or use column chromatographic clean-up with silica gel, alumina, or Florisil (6).

12.4.5 An electrochemical detector may be used to improve sensitivity for some ureas, carbamates, and phenolics. Much more care is required in using this detector, particularly in removing dissolved oxygen from the mobile phase and sample extracts.

12.4.6 Chlorophenol (di- through penta-) may be analyzed by GC/ECD or GC/MS after derivatization with pentafluorobenzylbromide (EPA Method 604).

12.4.7 Chlorinated phenoxyacetic acid herbicides and pentachlorophenol can be analyzed by GC/ECD or GC/MS after derivatization with diazomethane (EPA Method 515). DB-5 and DB-1701 columns (0.25-mm I.D. x 30-m) at 60 to 300°C/4°C per min have been found to perform well.

12.5 Analysis of Pesticides and PCBs by Gas Chromatography with Mass Spectrometry Detection (GC/MS)

[Note: A mass spectrometer operating in the selected ion monitoring mode is useful for confirmation and identification of pesticides.]

12.5.1 A mass spectrometer operating in the select ion monitoring (SIM) mode can be used as a sensitive detector for multi-residue determination of a wide variety of pesticides. Mass spectrometers are now available that provide detection limits comparable to nitrogen-phosphorus and electron capture detectors.

12.5.2 Most of the pesticides shown in Table 1 have been successfully determined by GC/MS/SIM. Typical GC operating parameters are as described in Section 12.1.1.

12.5.3 The mass spectrometer is typically operated using positive ion electron impact ionization (70 eV). Other instrumental parameters are instrument specific.

12.5.4 p-Terphenyl-d_{14} is commonly used as a surrogate for GC/MS analysis.

12.5.5 Quantification is typically performed using an internal standard method. 1,4-Dichlorobenzene, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12} and perylene-d_{12} are commonly used as internal standards. Procedures given in Section 12.1.1 through 12.1.9 and Section 12.1.13 through 12.1.14 apply, except for the selection of surrogates, detector, and make up gas.

12.5.6 See ASTM Practice D 3687 for injection technique, determination of relative retention times, and other procedures pertinent to GC and HPLC analyses.

12.6 Sample Concentration

12.6.1 If concentrations are too low to detect by the analytical procedure of choice, the extract may be concentrated to 1 mL or 0.5 mL by carefully controlled evaporation under an inert atmosphere. The following procedure is appropriate.

12.6.2 Place K-D concentrator tube in a water bath and analytical evaporator (nitrogen blow-down) apparatus. The water bath temperature should be from 25°C to 50°C.

12.6.3 Adjust nitrogen flow through hypodermic needle to provide a gentle stream.

12.6.4 Carefully lower hypodermic needle into the concentrator tube to a distance of about 1 cm above the liquid level.

12.6.5 Continue to adjust needle placement as liquid level decreases.

12.6.6 Reduce volume to slightly below desired level.

12.6.7 Adjust to final volume by carefully rinsing needle tip and concentrator tube well with solvent (usually n-hexane).

13. Calculations

13.1 Determination of Concentration

13.1.1 The concentration of the analyte in the extract solution can be taken from a standard curve where peak height or area is plotted linearly against concentration in nanograms per milliliter (ng/mL). If the detector response is known to be linear, a single point is used as a calculation constant.

13.1.2 From the standard curve, determine the nanograms of analyte standard equivalent to the peak height or area for a particular compound.

13.1.3 Ascertain whether the field blank is contaminated. Blank levels should not exceed 10 ng/sample for organochlorine pesticides or 100 ng/sample for PCBs and other pesticides. If the blank has been contaminated, the sampling series must be held suspect.

13.1.4 Quantity of the compound in the sample (A) is calculated using the following equation:

$$A = 1000 \left(\frac{A_s \times V_e}{V_i} \right)$$

where:

A = total amount of analyte in the sample, ng.

A_s = calculated amount of material injected onto the chromatograph based on calibration curve for injected standards, ng.

V_e = final volume of extract, mL.

V_i = volume of extract injected, μL.

1000 = factor for converting microliters to milliliters.

13.1.5 The extraction efficiency (EE) is determined from the recovery of surrogate spike as follows:

$$EE(\%) = \left| \frac{S}{S_a} \right| [100]$$

where:

EE = extraction efficiency, %.

S = amount of spike recovered, ng.

S_a = amount of spike added to plug, ng.

The extraction efficiency (surrogate recovery) must fall between 60-120% to be acceptable.

13.1.6 The total volume of air sampled under ambient conditions is determined using the following equation:

$$V_a = \frac{\sum_{i=1}^{n} (T_i \times F_i)}{1000 \text{ L/m}^3}$$

where:

V_a = total volume of air sampled, m^3.

T_i = length of sampling segment between flow checks, min.

F_i = average flow during sampling segment, L/min.

13.1.7 The air volume is corrected to EPA standard temperature (25°C) and standard pressure (760 mm Hg) as follows:

$$V_s = V_a \left(\frac{P_b - P_w}{760 \text{ mm Hg}} \right) \left(\frac{298K}{t_A} \right)$$

where:

V_s = volume of air at standard conditions (25°C and 760 mm Hg), std. m^3.

V_a = total volume of air sampled, m^3.

P_b = average ambient barometric pressure, mm Hg.

P_w = vapor pressure of water at calibration temperature, mm Hg.

t_A = average ambient temperature, °C + 273.

13.1.8 If the proper criteria for a sample have been met, concentration of the compound in a standard cubic meter of air sampled is calculated as follows:

$$C_a(\text{ng/std. m}^3) = \left[\frac{(A)}{(V_s)} \right] \left[\frac{(100)}{(SE(\%))} \right]$$

where:

SE = sampling efficiency as determined by the procedure outlined in Section 14.

If it is desired to convert the air concentration value to parts per trillion (ppt) in dry air at standard temperature and pressure (STP), the following conversion is used:

$$\text{ppt} = 0.844 \, (C_a)$$

The air concentration can be converted to parts per trillion (v/v) in air at STP as follows:

$$\text{pptv} = \left[\frac{(24.45) \, (C_a)}{(MW)} \right]$$

where:

MW = molecular weight of the compound of interest, g/g-mole.

13.1.9 If quantification is performed using an internal standard, a relative response factor (RRF) is calculated by the equation:

$$RRF = \left[\frac{(I_s)(C_{is})}{(I_{is})(C_s)}\right]$$

where:

I_s = integrated area of the target analyte peak, counts.
I_{is} = integrated area of the internal standard peak, counts.
C_{is} = concentration of the internal standard, ng/μL.
C_s = concentration of the analyte, ng/μL.

13.1.10 The concentration of the analyte (C_a) in the sample is then calculated as follows:

$$C_a = \frac{(I_s)(C_{is})}{(RRF)(I_{is})}$$

where:

C_a = concentration of analyte, ng/m^3
I_s = integrated area of the target analyte peak, counts.
RRF = relative response factor (see Section 13.1.10).

14. Sampling and Retention Efficiencies

14.1 General

14.1.1 Before using Compendium Method TO-10A, the user should determine the sampling efficiency for the compound of interest. The sampling efficiencies shown in Tables 2, 3, 4, and 5 were determined for approximately 1 m^3 of air at about 25°C, sampled at 3.8 L/min. The SE values in these tables may be used for similar sampling conditions; for other compounds or conditions, SE values must be determined.

14.1.2 Sampling efficiencies for the pesticides shown in Table 6 are for a flowrate of 3.8 L/min and at 25°C. For compounds not listed, longer sampling times, different flow rates, or other air temperatures, the following procedure may be used to determine sampling efficiencies.

14.2 Determining SE

14.2.1 SE is determined by a modified impinger assembly attached to the sampler pump, as illustrated in Figure 7. A clean PUF is placed in the pre-filter location and the inlet is attached to a nitrogen line.

[Note: Nitrogen should be used instead of air to prevent oxidation of the compounds under test. The oxidation would not necessarily reflect what may be encountered during actual sampling and may give misleading sampling efficiencies.]

Two PUF plugs (22-mm x 7.6-cm) are placed in the primary and secondary traps and are attached to the pump.

14.2.2 A standard solution of the compound of interest is prepared in a volatile solvent (i.e., hexane, pentane, or benzene). A small, accurately measured volume (i.e., 1 mL) of the standard solution is placed into the modified midget impinger. The sampler pump is set at the rate to be used in field application and then activated. Nitrogen is drawn through the assembly for a period of time equal to or exceeding that intended for field application. After the desired sampling test period, the PUF plugs are removed and analyzed separately as per Section 12.

14.2.3 The impinger is rinsed with hexane or another suitable solvent and quantitatively transferred to a volumetric flask or concentrator tube for analysis.

14.2.4 The sampling efficiency (SE) is determined using the following equation:

$$\% \text{ SE} = \frac{W_1}{W_0 - W_r} \times 100$$

where:

W_1 = amount of compound extracted from the primary trap, ng.

W_o = original amount of compound added to the impinger, ng.

W_r = residue left in the impinger at the end of the test, ng.

14.2.5 If material is found in the secondary trap, it is an indication that breakthrough has occurred. The addition of the amount found in the secondary trap, W_2, to W_1, will provide an indication for the overall sampling efficiency of a tandem-trap sampling system. The sum of W_1, W_2 (if any), and W_r must equal (approximately ±10%) W_o or the test is invalid.

14.2.6 If the compound of interest is not sufficiently volatile to vaporize at room temperature, the impinger may be heated in a water bath or other suitable heater to a maximum of 50°C to aid volatilization. If the compound of interest cannot be vaporized at 50°C without thermal degradation, dynamic retention efficiency (RE_d) may be used to estimate sampling efficiency. Dynamic retention efficiency is determined in the manner described in Section 14.2.7. Table 7 lists those organochlorine pesticides which dynamic retention efficiencies have been determined.

14.2.7 A pair of PUF plugs is spiked by slow, dropwise addition of the standard solution to one end of each plug. No more than 0.5 to 1 mL of solution should be used. Amounts added to each plug should be as nearly the same as possible. The plugs are allowed to dry for 2 hours in a clean, protected place (i.e., desiccator). One spiked plug is placed in the primary trap so that the spiked end is at the intake and one clean unspiked plug is placed in the secondary trap. The other spiked plug is wrapped in hexane-rinsed aluminum foil and stored in a clean place for the duration of the test (this is the static control plug, Section 14.2.8). Prefiltered nitrogen or ambient air is drawn through the assembly as per Section 14.2.2.
[Note: Impinger may be discarded.]

Each PUF plug (spiked and static control) is analyzed separately as per Section 12.

14.2.8 This dynamic retention efficiency (% RE_d) is calculated as follows:

$$\% \text{ RE}_d = \frac{W_1}{W_o} \times 100$$

where:

W_1 = amount of compound recovered from primary plug, ng.

W_o = amount of compound added to primary plug, ng.

If a residue, W_2, is found on the secondary plug, breakthrough has occurred. The sum of $W_1 + W_2$ must equal W_o, within 25% or the test is invalid. For most compounds tested by this procedure, % RE_d values are generally less than % SE values determined per Section 14.2. The purpose of the static RE_d determination is to establish any loss or gain of analyte unrelated to the flow of nitrogen or air through the PUF plug.

15. Performance Criteria and Quality Assurance

[Note: This section summarizes required quality assurance (QA) measures and provides guidance concerning performance criteria that should be achieved within each laboratory.]

15.1 Standard Operating Procedures (SOPs)

15.1.1 Users should generate SOPs describing the following activities accomplished in their laboratory: (1) assembly, calibration, and operation of the sampling system, with make and model of equipment used; (2) preparation, purification, storage, and handling of sampling cartridges; (3) assembly, calibration, and operation of the analytical system, with make and model of equipment used; and (4) all aspects of data recording and processing, including lists of computer hardware and software used.

15.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by, the laboratory personnel conducting the work.

15.2 Process, Field, and Solvent Blanks

15.2.1 One PUF cartridge from each batch of approximately twenty should be analyzed, without shipment to the field, for the compounds of interest to serve as a process blank.

15.2.2 During each sampling episode, at least one PUF cartridge should be shipped to the field and returned, without drawing air through the sampler, to serve as a field blank.

15.2.3 Before each sampling episode, one PUF plug from each batch of approximately twenty should be spiked with a known amount of the standard solution. The spiked plug will remain in a sealed container and will not be used during the sampling period. The spiked plug is extracted and analyzed with the other samples. This field spike acts as a quality assurance check to determine matrix spike recoveries and to indicate sample degradation.

15.2.4 During the analysis of each batch of samples, at least one solvent process blank (all steps conducted but no PUF cartridge included) should be carried through the procedure and analyzed.

15.2.5 All blank levels should not exceed 10 ng/sample for single components or 100 ng/sample for multiple component mixtures (i.e., for organochlorine pesticides and PCBs).

15.3 Sampling Efficiency and Spike Recovery

15.3.1 Before using the method for sample analysis, each laboratory must determine its sampling efficiency for the component of interest as per Section 14.

15.3.2 The PUF in the sampler is replaced with a hexane-extracted PUF. The PUF is spiked with a microgram level of compounds of interest by dropwise addition of hexane solutions of the compounds. The solvent is allowed to evaporate.

15.3.3 The sampling system is activated and set at the desired sampling flow rate. The sample flow is monitored for 24 hours.

15.3.4 The PUF cartridge is then removed and analyzed as per Section 12.

15.3.5 A second sampler, unspiked, is collected over the same time period to account for any background levels of components in the ambient air matrix.

15.3.6 In general, analytical recoveries and collection efficiencies of 75% are considered to be acceptable method performance.

15.3.7 Replicate (at least triplicate) determinations of collection efficiency should be made. Relative standard deviations for these replicate determinations of ±15% or less are considered acceptable performance.

15.3.8 Blind spiked samples should be included with sample sets periodically as a check on analytical performance.

15.4 Method Precision and Bias

15.4.1 Precision and bias in this type of analytical procedure are dependent upon the precision and bias of the analytical procedure for each compound of concern, and the precision and bias of the sampling process.

15.4.2 Several different parameters involved in both the sampling and analysis steps of this method collectively determine the precision and bias with which each compound is detected. As the volume of air sampled is increased, the sensitivity of detection increases proportionately within limits set by: (a) the retention efficiency for each specific component trapped on the polyurethane foam plug, and (b) the background interference associated with the analysis of each specific component at a given site sampled. The sensitivity of detection of samples recovered by extraction depends on: (a) the inherent response of the particular GC detector used in the determinative step, and (b) the extent to which the sample is concentrated for analysis. It is the responsibility of the analyst(s) performing the sampling and analysis steps to adjust parameters so that the required detection limits can be obtained.

15.4.3 The reproducibility of this method for most compounds for which it has been evaluated has been determined to range from ±5 to ±30% (measured as the relative standard deviation) when replicate sampling cartridges are used (N>5). Sample recoveries for individual compounds generally fall within the range of 90 to 110%, but recoveries ranging from 65 to 125% are considered acceptable. PUF alone may give lower recoveries for more volatile compounds (i.e., those with saturation vapor pressures $>10^{-3}$ mm Hg). In those cases, another sorbent or a combination of PUF and Tenax TA (see Figure 2) should be employed.

15.5 Method Safety

15.5.1 This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use.

15.5.2 It is the user's responsibility to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

16. References

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TABLE 1. COMPOUNDS FOR WHICH PROCEDURE HAS BEEN TESTED[1]

Compound	Recommended Analysis[2]	Compound	Recommended Analyses
Alachlor	GC/ECD	Heptachlor	GC/ECD
Aldrin	GC/ECD	Heptachlor epoxide	GC/ECD
Allethrin	HPLC/UV	Hexachlorobenzene	GC/ECD
Aroclor 1242	GC/ECD	Hexachlorocyclopentadiene[3,4]	GC/ECD
Aroclor 1254	GC/ECD	Lindane (γ-BHC)	GC/ECD
Aroclor 1260	GC/ECD	Linuron	HPLC/UV
Atrazine	GC/NPD	Malathion	GC/NPD or FPD
Bendiocarb	HPLC/UV	Methyl parathion	GC/NPD or FPD
BHC (α- and β-Hexachlorocyclohexanes)	GC/ECD	Methoxychlor	GC/FCD
Captan	GC/ECD	Metolachlor	GC/ECD
Carbaryl	HPLC/UV	Mexacarbate	GC/FCD
Carbofuran	HPLC/UV	Mirex	GC/ECD
Chlordane, technical	GC/ECD	Monuron	HPLC/UV
Chlorothalonil	GC/ECD	Trans-nonachlor	GC/ECD
Chlorotoluron	HPLC/UV	Oxychlordane	GC/ECD
Chlorpyritos	GC/ECD	Pentachlorobenzene	GC/ECD
2,4-D esters and salts	GC/ECD	Pentachlophenol	GC/ECD
Dacthal	GC/ECD	Permethrin (cis and trans)	HPLC/UV
ρ,ρ-'DDT	GC/ECD	o-Phenylphenol	HPLC/UV
ρ,ρ-'DDE	GC/ECD	Phorate	GC/NPD or FPD
Diazinon	GC/NPD or FPD	Propazine	GC/NPD
Dicloran	GC/ECD	Propoxur (Baygon)	HPLC/UV
Dieldrin	GC/ECD	Pyrethrin	HPLC/UV
Dichlorovos (DDVP)	GC/ECD	Resmethrin	HPLC/UV
Dicofol	GC/ECD	Ronnel	GC/ECD
Dicrotophos	HPLC/UV	Simazine	HPLC/UV
Diuron	HPLC/UV	Terbuthiuron	HPLC/UV
Ethyl parathion	GC/NPD or FPD	1,2,3,4-tetrachlorobenzene[3]	GC/ECD
Fenvalerate	HPLC/UV	1,2,3-trichlorobenzene[3]	GC/ECD
Fluometuron	HPLC/UV	2,3,5-trichlorophenol	GC/ECD
Folpet	GC/ECD	Trifluralin	GC/ECD

[1]The following recommendations are specific for that analyte for maximum sensitivity.
[2]GC = gas chromatography; ECD = electron capture detector, FPD = flame photometric detector; HPLC = high performance liquid chromatography; NPD = nitrogen-phosphorus detector; UV = ultraviolet absorption detector, (GC/MS (gas chromatography/mass spectrometry) may also be used).
[3]Using PUF/Tenax-TA "sandwich" trap.
[4]Compound is very unstable in solution.

TABLE 2. SAMPLING EFFICIENCIES FOR SOME ORGANOCHLORINE PESTICIDES

Compound	Quantity Introduced, μg^2	Air Volume, m^3	Sampling efficiency, %		
			mean	RSD	n
α-Hexachlorocyclohexane (α-BHC)	0.005	0.9	115	8	6
γ-Hexachlorocyclohexane (Lindane)	0.05-1.0	0.9	91.5	8	5
Chlordane, technical	0.2	0.9	84.0	11	8
p,p'-DDT	0.6, 1.2	0.9	97.5	21	12
p,p'-DDE	0.2, 0.4	0.9	102	11	12
Mirex	0.6, 1.2	0.9	85.9	22	7
2,4-D Esters:					
Isopropyl	0.5	3.6	92.0	5	12
Butyl	0.5	3.6	82.0	10	11
Isobutyl	0.5	3.6	79.0	20	12
Isoctyl	0.5	3.6	>80[2]	--	--

[1]Air volume = 0.9 m^3.
[2]Not vaporized. Value base on %RE = 81.0 (RSD = 10%, n = 6).

TABLE 3. SAMPLING EFFICIENCIES FOR ORGANOPHOSPHORUS PESTICIDES

Compound	Quantity Introduced, μg^2	Sampling efficiency, %		
		mean	RSD	n
Dichlorvos (DDVP)	0.2	72.0	13	2
Ronnel	0.2	106	8	12
Chlorpyrifos	0.2	108	9	12
Diazinon[1]	1.0	84.0	18	18
Methyl parathion[1]	0.6	80.0	19	18
Ethyl parathion[1]	0.3	75.9	15	18
Malathion[1]	0.3	100[3]	--	--

[1]Analyzed by gas chromatography with nitrogen phosphorus detector or flame photometric detector.
[2]Air volume = 0.9 m^3.
[3]Decomposed in generator; value based on %RE = 101 (RDS = 7, n = 4).

TABLE 4. SAMPLING EFFICIENCIES FOR SOME SEMI-VOLATILE ORGANOCHLORINE COMPOUNDS AND PCBs

Compound	Quantity Introduced, μg[1]	Sampling efficiency, %		
		mean	RSD	n
1,2,3-Trichlorobenzene	1.0	6.6[2]	22	8
1,2,3,4-Tetrachlorobenzene	1.0	62.3[2]	33	5
Pentachlorobenzene	1.0	94.0	12	5
Hexachlorobenzene	0.5, 1.0	94.5	8	5
Hexachlorocyclopentadiene	1.0	8.3[2]	12	5
2,4,5-Trichlorophenol	1.0	108	3	5
Pentachlorophenol	1.0	107	16	5
Aroclor 1242	0.1	96.0	15	6
Aroclor 1254	0.1	95.0	7	6
Aroclor 1260	0.1	109	5	11

[1]Air volume = 0.9 m^3.
[2]% SEs were 98, and 97% (n = 2), respectively, for these three compounds by the PUF/Tenax® TA "sandwich" trap.

TABLE 5. SAMPLING EFFICIENCIES FOR CARBAMATES, UREAS, TRIAZINES, AND PYRETHRINS

Compound	Spike Level, µg/plug	Static Recovery, %			Retention Efficiency, %			Sampling Efficiency, %		
		mean	RSDP	n	mean	RSD	n	mean	RSD	n
Carbamates:										
Propoxur	5	61.4	10	6	77.6	37	6	96.7	11	6
Carbofuran	15	55.3	12	6	64.2	46	6	87.2	14	6
Bendicarb	50	57.3	11	6	69.8	43	6	62.1	14	6
Mexacarbate	10	62.8	19	6	62.7	41	6	89.8	14	6
Carbaryl	100	56.6	14	6	63.6	53	6	0	13	6
Ureas:										
Monuron	19	87.0	6	6	91.2	6	5	0		
Diuron	20	84.1	8	6	90.0	2	5	0		
Linuron	20	86.7	8	6	92.5	4	5	0		
Terbuthiuron	18	85.0	8	6	88.8	8	5	0		
Fluometuron	20	91.4	10	6	101	3	5	0		
Chlortoluron	20	86.2	11	6	92.0	7	5	0		
Triazines:										
Simazine	10	103	6	5	101	9	6	0		
Atrazine	10	104	7	5	98.9	7	6	0		
Propazine	10	105	11	5	99.9	14	6	0		
Pyrethrins:										
Pyrethrin I	(9.7)	90.5	10	6	95.6	22	5	0		
Pyrethrin II	(6.1)	88.6	11	6	69.9	29	5	0		
Allethrin	25	69.2	9	5	58.3	12	6	0		
d-trans-Allethrin	25	76.8	9	6	74.4	9	5	0		
Dicrotophos	25	72.0	22	6	71.7	8	5	0		
Resmethrin	25	76.5	14	6	66.7	14	6	0		
Fenvalerate	25	87.9	3	6	57.2	20	3	0		

TABLE 6. EXTRACTION AND 24-H SAMPLING EFFICIENCIES FOR VARIOUS PESTICIDES AND RELATED COMPOUNDS

Compound	Extraction Efficiency, %		Sampling Eficiency, %, at					
			10 ng/m³		100 ng/m³		1,000 ng/m³	
	mean	RSD	mean	RSD	mean	RSD	mean	RSD
Chlropyrifos	83.3	11.5	83.7	18.0	92.7	15.1	83.7	18.0
Pentachlorophenol	84.0	22.6	66.7	42.2	52.3	36.2	66.7	42.2
Chlordane	95.0	7.1	96.0	1.4	74.0	8.5	96.0	1.4
o-Phenylphenol	47.0	46.7	46.0	19.1	45.3	29.9	46.0	19.1
Lindane	96.0	6.9	91.7	11.6	93.0	2.6	91.7	11.6
DDVP	88.3	20.2	51.0	53.7	106.0	1.4	51.0	53.7
2,4-D Methyl Ester	--	--	75.3	6.8	58.0	23.6	75.3	6.8
Heptachlor	99.0	1.7	97.3	13.6	103.0	17.3	97.3	13.6
Aldrin	97.7	4.0	90.7	5.5	94.0	2.6	90.7	5.5
Dieldrin	95.0	7.0	82.7	7.6	85.0	11.5	82.7	7.6
Ronnel	80.3	19.5	74.7	12.1	60.7	15.5	74.7	12.2
Diazinon	72.0	21.8	63.7	18.9	41.3	26.6	63.7	19.9
trans-Nonachlor	97.7	4.0	96.7	4.2	101.7	15.3	96.7	4.2
Oxychlorodane	100.0	0.0	95.3	9.5	94.3	1.2	95.3	9.5
α-BHC	98.0	3.5	86.7	13.7	97.0	18.2	86.7	13.7
Bendiocarb	81.3	8.4	59.7	16.9	30.7	23.5	59.7	16.9
Chlorothalonil	90.3	8.4	76.7	6.1	70.3	6.5	76.7	6.1
Heptachlor Epoxide	100.0	0.0	95.3	5.5	97.7	14.2	95.3	5.5
Dacthal	--	--	87.0	9.5	95.3	22.2	87.0	9.5
Aroclor 1242	91.7	14.4	95.0	15.5	94.7	17.5	95.0	15.5

[1]Mean values for one spike at 550 ng/plug and two spikes at 5,500 ng/plug.
[2]Mean values for three determinations.

TABLE 7. EXTRACTION AND 24-H DYNAMIC RETENTION EFFICIENCIES FOR VARIOUS PESTICIDES AND RELATED COMPOUNDS

Compound	Extraction Efficiency, %		Sampling Eficiency, %, at					
			10 ng/m³		100 ng/m³		1,000 ng/m³	
	mean	RSD	mean	RSD	mean	RSD	mean	RSD
Propoxur	77.5	71.4	92.0	--	91.7	22.8	101.0	18.4
Resmethrin	95.5	71.4	79.0	--	100.7	13.1	107.0	4.4
Dicofol	57.0	8.5	38.0	25.9	65.0	8.7	69.0	--
Captan	73.0	12.7	56.0	--	45.5	64.3	84.3	16.3
Carbaryl	74.0	82.0	102.0	--	61.0	--	113.0	6.1
Malathion	76.5	44.5	108.0	--	54.0	16.0	77.3	7.6
cis-Permethrin	88.7	10.3	101.0	28.5	85.0	26.9	89.0	11.3
trans-Permethrin	88.7	11.0	67.3	34.8	80.7	56.4	108.3	9.5
Methoxychlor	65.5	4.9	--	--	--	--	78.5	2.1
Atrazine	75.0	50.5	--	--	73.0	30.1	83.0	9.5
Folpet	86.7	11.7	--	--	78.0	--	93.0	--
Aroclor 1260	92.0	14.5	88.0	9.6	85.3	9.9	107.1	13.6

[1]Mean values for one spike at 550 ng/plug and two spikes at 5,500 ng/plug.
[2]Mean values for three determinations.

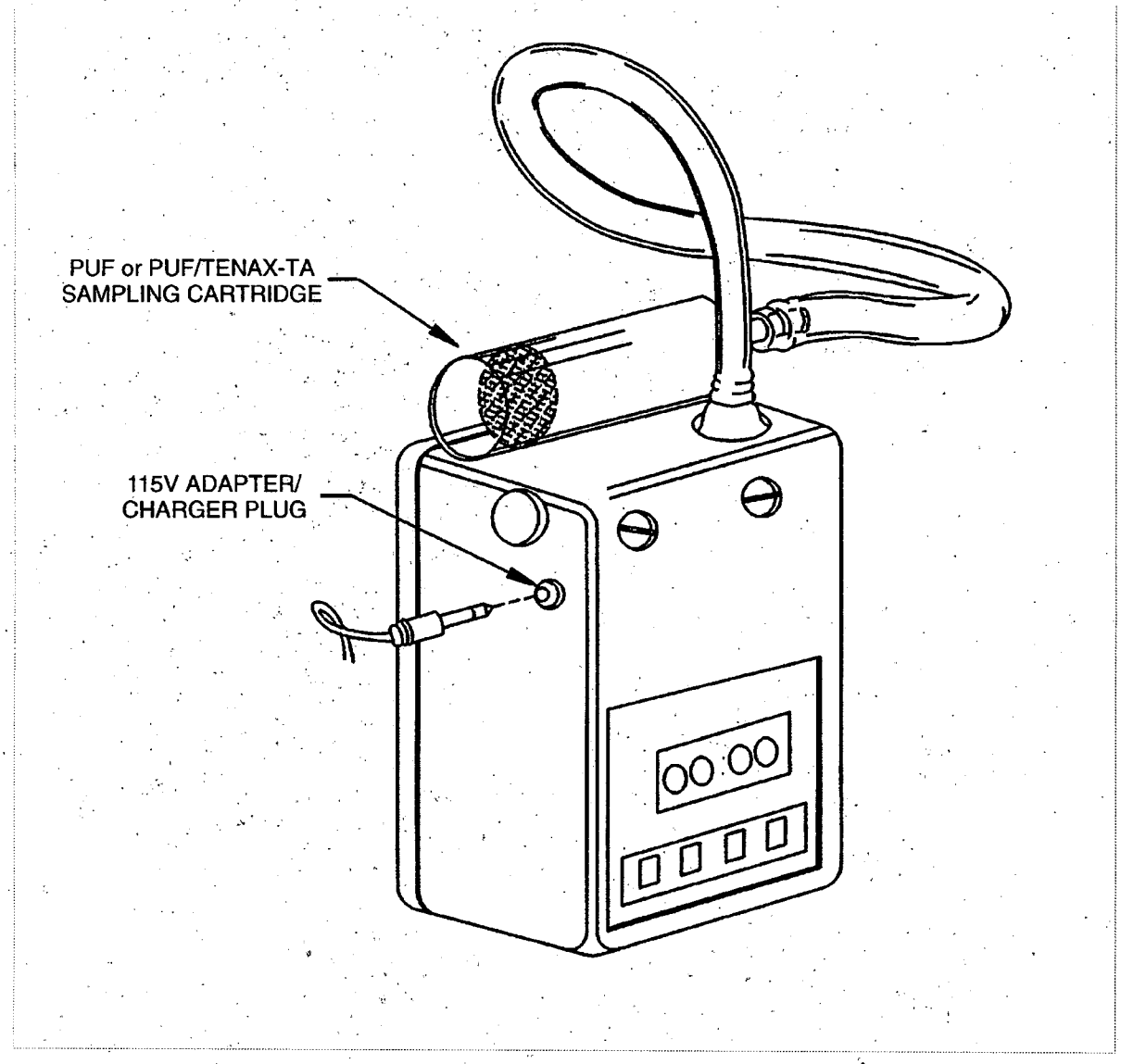

Figure 1. Low volume air sampler.

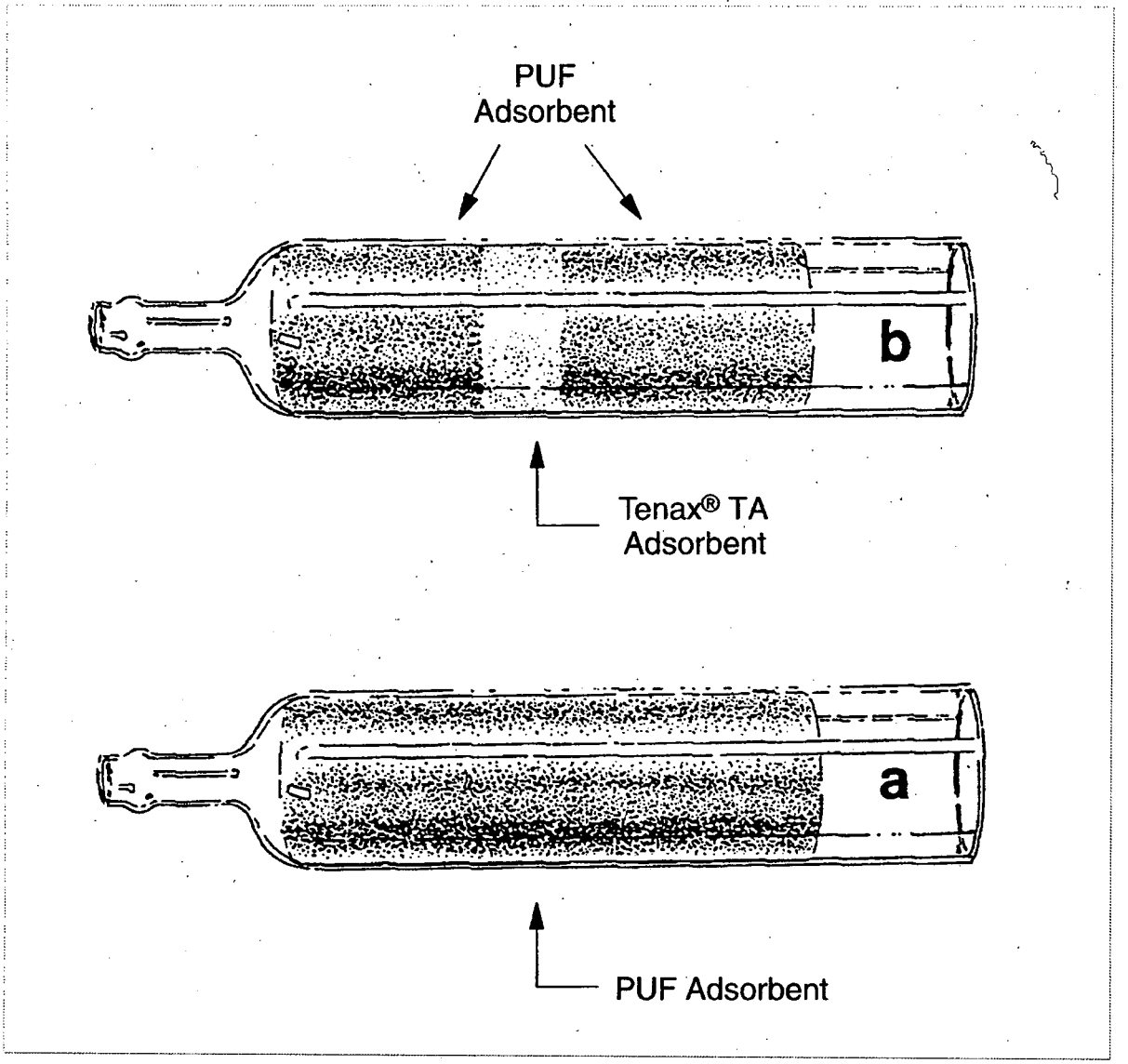

Figure 2. Polyurethane foam (PUF) sampling cartridge (a) and PUF-Tenax® TA "sandwich" sampling cartridge (b).

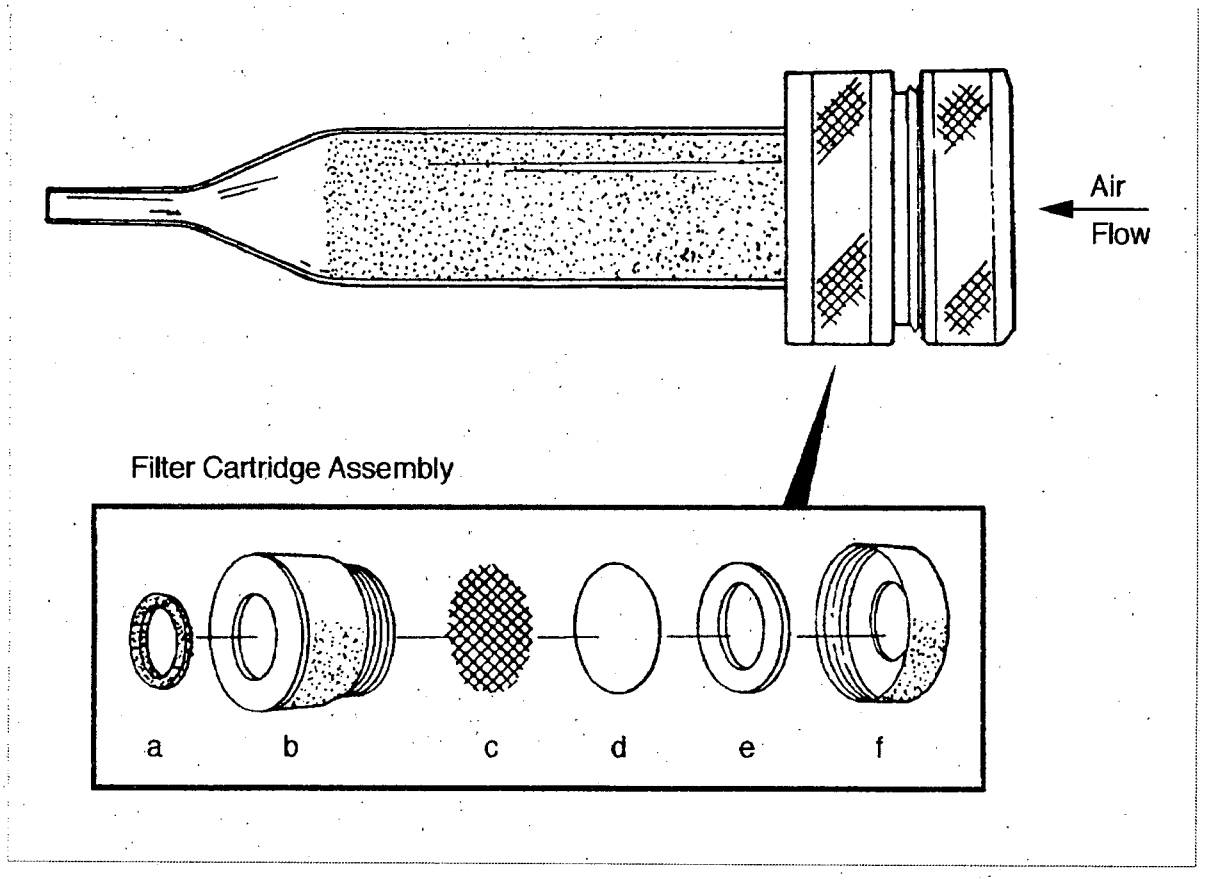

Figure 3. Open-face filter assembly attached to a PUF cartridge:
(a) Inner Viton® o-ring, (b) filter cartridge, (c) stainless steel screen, (d) quartz filter, (e) filter ring, and (f) cartridge screw cap.

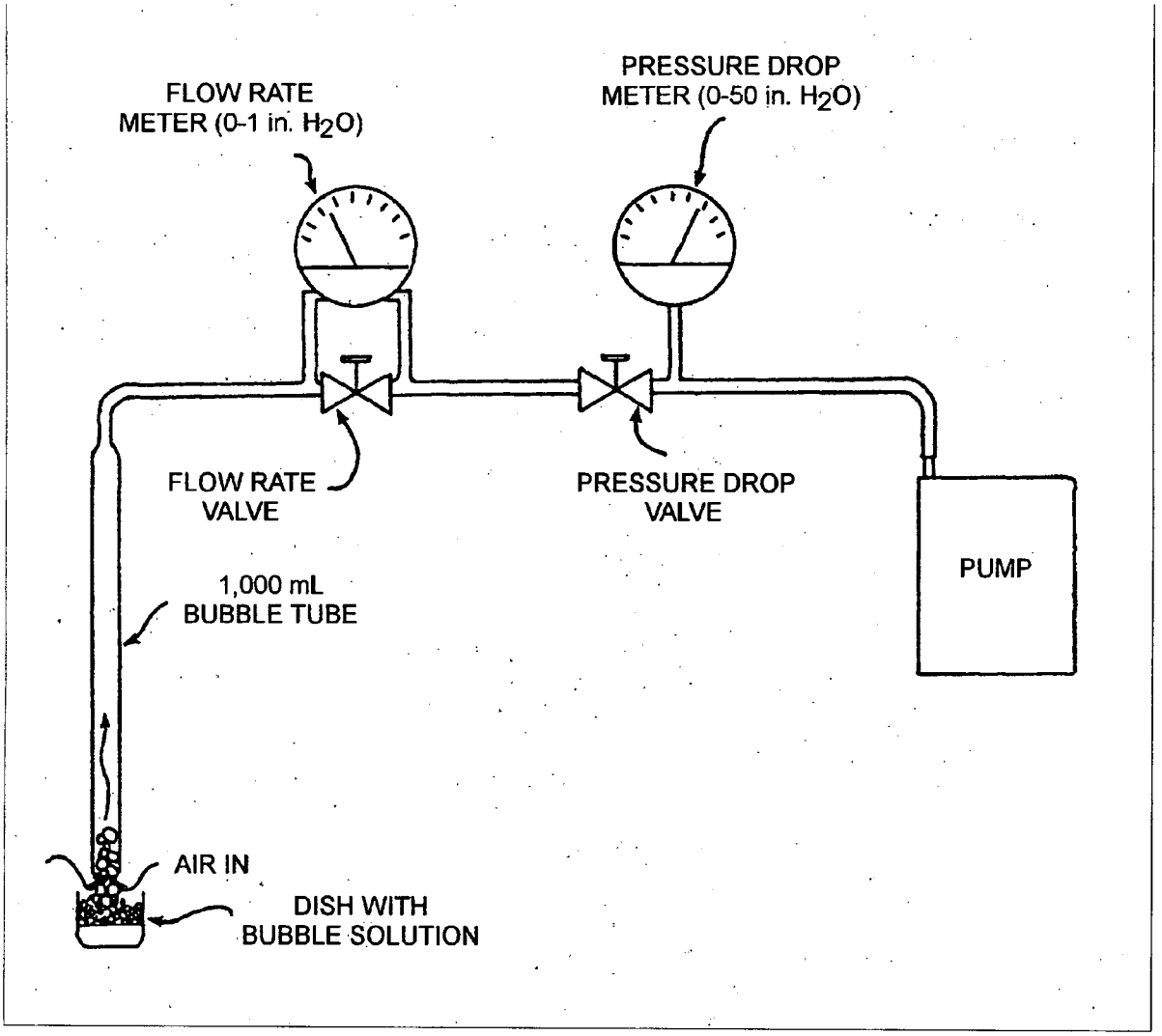

Figure 4. Calibration assembly for air sampler pump.

COMPENDIUM METHOD TO-10A
FIELD TEST DATA SHEET (FTDS)

I. GENERAL INFORMATION

PROJECT:_____ DATE(S) SAMPLED:_____
SITE:_____ TIME PERIOD SAMPLED:_____
LOCATION:_____ OPERATOR:_____
INSTRUMENT MODEL NO.:_____ CALIBRATED BY:_____
PUMP SERIAL NO.:_____ RAIN: ____YES _____NO

ADSORBENT CARTRIDGE INFORMATION:

	Cartridge 1	Cartridge 2	Cartridge 3	Cartridge 4
Type:				
Adsorbent:				
Serial No.:				
Sample No.:				

II. SAMPLING DATA

Cartridge Identification	Sampling Location	Ambient Temp., °F	Ambient Pressure, in Hg	Flow Rate (Q), mL/min		Sampling Period		Total Sampling Time, min.	Total Sample Volume, L
				Cartridge 1	Cartridge 2	Start	Stop		

III. FIELD AUDIT

	Cartridge 1	Cartridge 2	Cartridge 3	Cartridge 4
Audit Flow Check Within 10% of Set Point (Y/N)?	___	___	___	___
	pre-	pre-	pre-	pre-
	post-	post-	post-	post-

CHECKED BY:_____

DATE:_____

Figure 5. Compendium Method TO-10A field test data sheet.

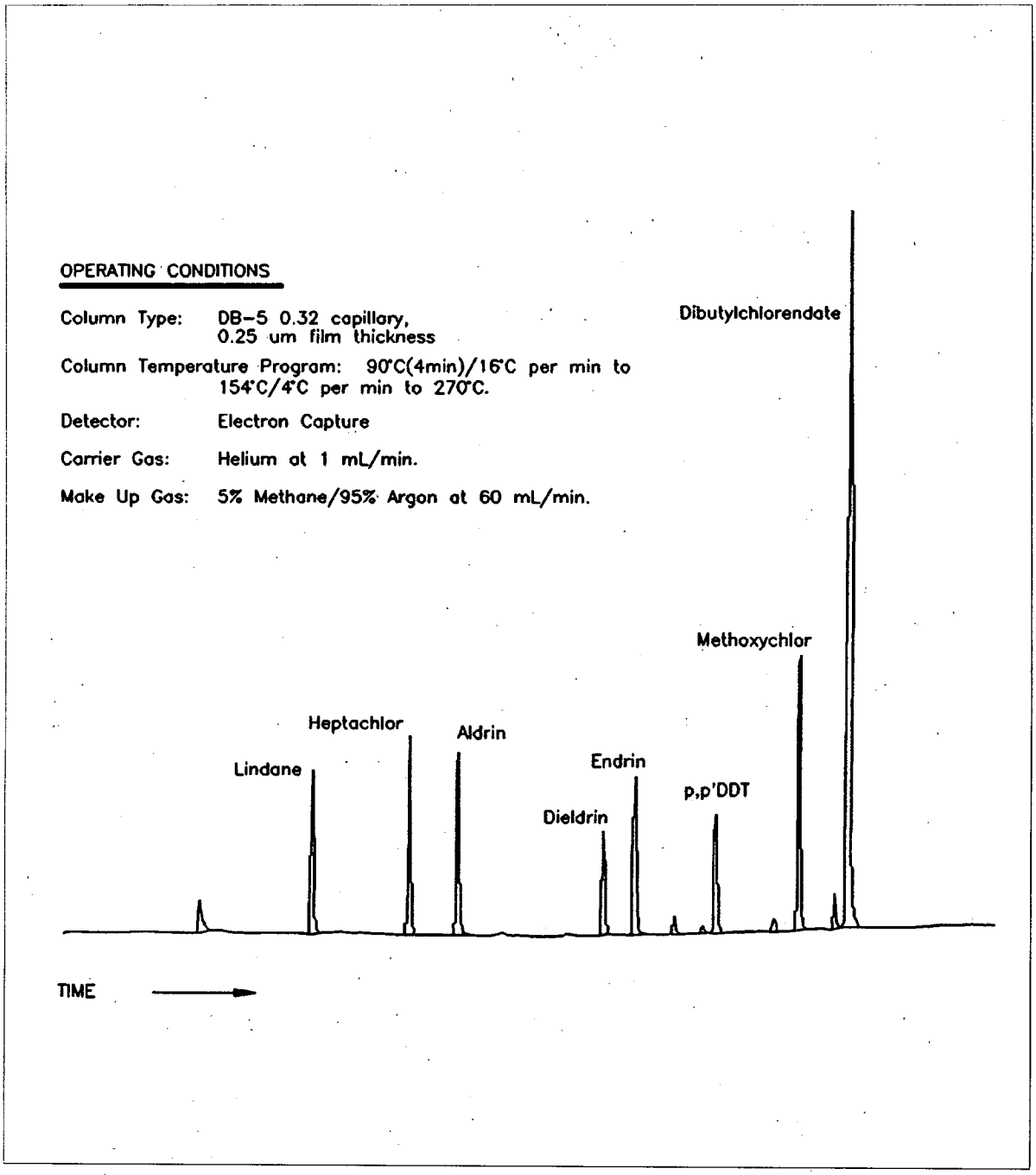

Figure 6. Chromatogram showing a mixture of single component pesticides determined by GC/ECD using a capillary column.

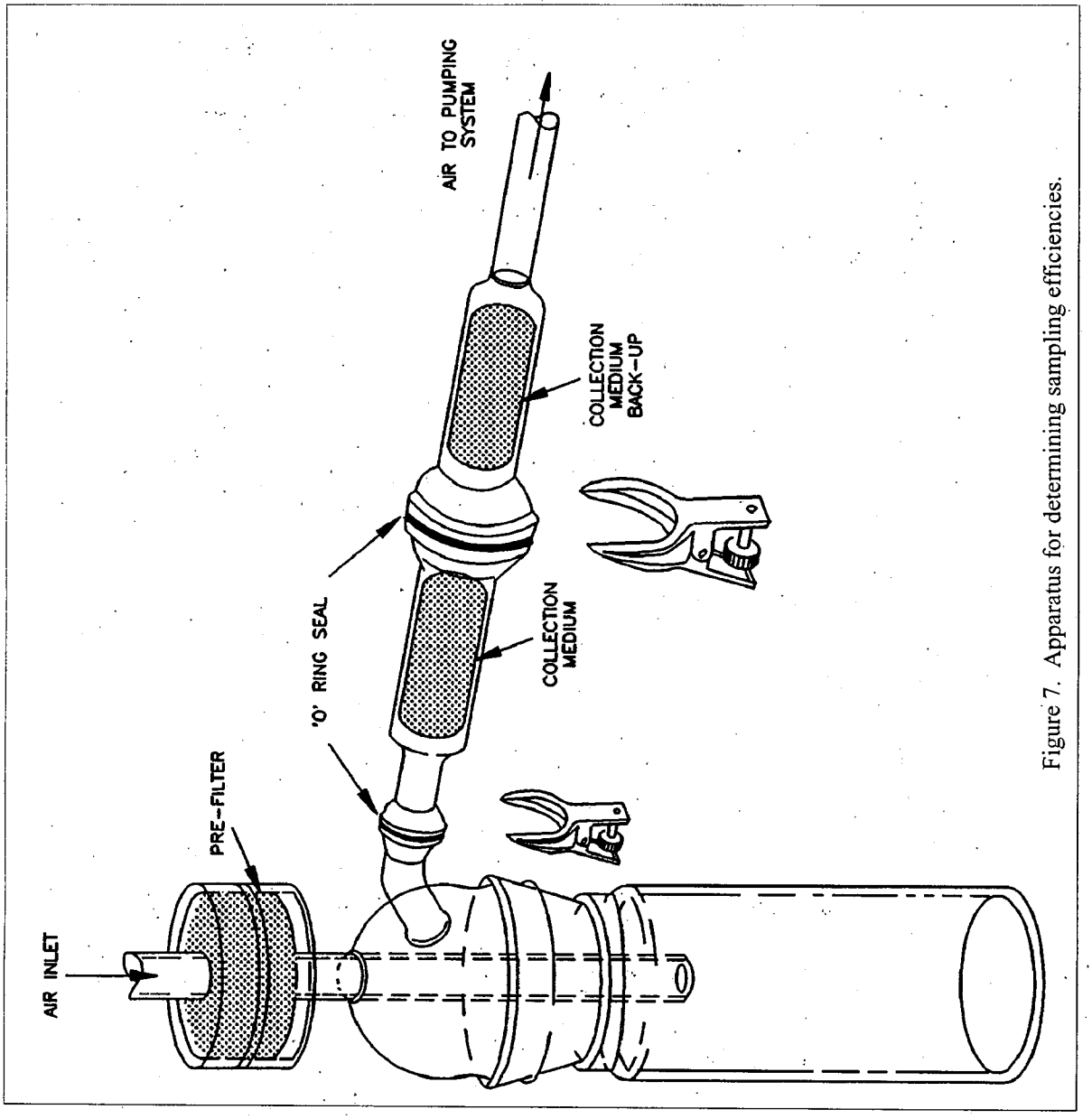

Figure 7. Apparatus for determining sampling efficiencies.

ATTACHMENT B

EPA/ERT SOP No. 2008 - General Air Sampling

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) provides guidance in developing and implementing sampling plans to assess the impact of hazardous waste sites on ambient air. It presents the United States Environmental Protection Agency/Environmental Response Team's (U.S. EPA/ERT's) approach to air sampling and monitoring and identifies equipment requirements. It is not within the scope of this SOP to provide a generic air sampling plan. Experience, objectives, site characteristics, and chemical characteristics will dictate sampling strategy. This SOP does not address indoor air sampling.

Two basic approaches can be used to assess ambient air (also referred to as air pathway assessments): modeling and measurements. The modeling approach initially estimates or measures the overall site emission rate(s) and pattern(s). These data are input into an appropriate air dispersion model, which predicts either the maximum or average air concentrations at selected locations or distances during the time period of concern. This overall modeling strategy is presented in the first three volumes of the Air Superfund National Technical Guidance Series on Air Pathway Assessments[1,2,3]. Specific applications of this strategy are presented in several additional Air Superfund Technical Guidance documents[4].

The measurement approach involves actually measuring the air impact at selected locations during specific time periods. These measurements can be used to document actual air impacts during specific time intervals (i.e., during cleanup operations) or to extrapolate the probable "worst case" concentrations at that and similar locations over a longer time period than was sampled.

This SOP addresses issues associated with this second assessment strategy. This SOP also discusses the U.S. EPA/ERT's monitoring instruments, air sampling kits, and approach to air sampling and monitoring at hazardous waste sites.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, depending on site conditions, equipment limitations, or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

2.0 METHOD SUMMARY

Air monitoring is defined as the use of direct-reading instruments and other screening or monitoring equipment and techniques that provide instantaneous (real-time) data on the levels of airborne contaminants. The U.S. EPA/ERT maintains numerous monitors for real-time measurements. Examples of air monitoring equipment are hand-held photoionization detectors (PID), flame ionization detectors (FID), oxygen/combustible gas detectors, and remote optical sensors.

Air sampling is defined as those sampling and analytical techniques that require either off- or on-site laboratory analysis and therefore do not provide immediate results. Typically, air sampling occurs after use of real-time air monitoring equipment has narrowed the number of possible contaminants and has provided some qualitative measurement of contaminant concentration. Air sampling techniques are used to more accurately detect, identify and quantify specific chemical compounds relative to the majority of air monitoring technologies.

In the Superfund Removal Program, On-Scene Coordinators (OSCs) may request the U.S. EPA/ERT to conduct air monitoring and sampling during the

following situations: emergency responses, site assessments, and removal activities. Each of these activities has a related air monitoring/sampling objective that is used to determine the potential hazards to workers and/or the community.

- Emergency Response

  Emergency responses are immediate responses to a release or threatened release of hazardous substances presenting an imminent danger to public health, welfare, or the environment (i.e., chemical spills, fires, or chemical process failures resulting in a controlled release of hazardous substances). Generally these situations require rapid on-site investigation and response. A major part of this investigation consists of assessing the air impact of these releases.

- Removal Site Assessment

  Removal site assessments (referred to as site assessments) are defined as any of several activities undertaken to determine the extent of contamination at a site and which help to formulate the appropriate response to a release or threatened release of hazardous substances. These activities may include a site inspection, multimedia sampling, and other data collection.

- Removal Actions

  Removal actions clean up or remove hazardous substances released into the environment. Removal actions include any activity conducted to abate, prevent, minimize, stabilize, or eliminate a threat to public health or welfare, or to the environment.

Personal risk from airborne contaminants can be determined by comparing the results of on-site monitoring and sampling to health-based action levels such as the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) and the Occupational Safety and Health Administration (OSHA) Permissible Exposure Limits (PELs). Residential risk can be determined by comparing the results of off-site monitoring or sampling to health-based action levels such as those developed by the Agency for Toxic Substance and Disease Registry (ATSDR).

The extent to which valid inferences can be drawn from air monitoring/sampling depends on the degree to which the monitoring/sampling effort conforms to the objectives of the event. Meeting the project's objectives requires thorough planning of the monitoring/sampling activities, and implementation of the most appropriate monitoring/sampling and analytical procedures. These issues will be discussed in this SOP.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Preservation, containers, handling and storage for air samples are discussed in the specific SOPs for the technique selected. In addition, the analytical method (i.e., U.S. EPA, National Institute for Occupational Safety and Health [NIOSH], and OSHA Methods) may be consulted for storage temperature, holding times and packaging requirements. After sample collection, the sampling media (i.e., cassettes or tubes) are immediately sealed. The samples are then placed into suitable containers (i.e., whirl bags, resealable bagsor culture tubes) which are then placed into a shipping container.

Use bubble wrap or styrofoam peanuts when packing air samples for shipment. DO NOT USE VERMICULITE.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Upwind sources can contribute to sample concentration. Natural sources, such as biological waste, can produce hydrogen sulfide and methane which may contribute to the overall contaminant level. Extraneous anthropogenic contaminants (i.e., burning of fossil fuels; emissions from vehicular traffic, especially diesel; volatile compounds from petrochemical facilities; and effluvium from smoke stacks) may also contribute. Air sampling stations should be strategically placed to identify contributing sources.

Photoreactivity or reaction of the parameters of concern may occur with nonrelated compounds [i.e., nitrogen compounds and polyaromatic hydrocarbons

(PAHs)]. Some sorbent media/samples should not be exposed to light during or after sampling due to photochemical effects (i.e., PAHs).

Various environmental factors, including humidity, temperature and pressure, also impact the air sampling methodology, collection efficiency and detection limit. Since the determination of air contaminants is specifically dependent on the collection parameters and efficiencies, the collection procedure is an integral part of the analytical method.

Detection limits depend on the contaminants being investigated and the particular site situation. It is important to know why the data are needed and how the data will be used. Care should be taken to ensure the detection limits are adequate for the intended use of the final results.

Some equipment may be sensitive to humidity and temperature extremes.

5.0 EQUIPMENT/APPARATUS

5.1 Direct Reading Instruments (Air Monitoring Instruments)

There are two general types of direct reading instruments: portable screening devices and specialized analytical instruments. Generally all these techniques involve acquiring, for a specific location or area, continuous or sequential direct air concentrations in either a real-time or semi-real-time mode. None of these instruments acquires true time-weighted average concentrations. In addition, these instruments are not capable of acquiring simultaneous concentration readings at multiple locations, although several are able to sequentially analyze samples taken remotely from different locations. The document, "Guide to Portable Instruments for Assessing Airborne Pollutants Arising from Hazardous Waste Sites[5]," provides additional information about air sampling and monitoring. The hazard levels for airborne contaminants vary. See the ACGIH TLVs and the OSHA PELs for safe working levels. Common screening devices and analytical instruments are described in Appendix A.

5.2 Air Sampling Equipment and Media/Devices

The U.S. EPA/ERT uses the following analytical methods for sampling: *NIOSH Manual of Analytical Methods*[6], *American Society for Testing and Materials (ASTM) Methods*[7], *U.S. EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air*[8,9], and *OSHA Methods*[10]. Additional air sampling references include *Industrial Hygiene and Toxicology* (3rd Ed.)[11] and *Air Sampling Instruments for Evaluation of Atmospheric Contaminants*[12]. These methods typically specify equipment requirements for sampling. Since air sampling is such a diverse technology, no single method or reference is best for all applications. Common sampling equipment and media/devices are described in Appendix B.

5.3 Tools/Material and Equipment List

In addition to equipment and materials identified in Appendices A and B, the following equipment and materials may be required to conduct air sampling and monitoring at hazardous waste sites:

- Camera
- Site logbook
- Clipboard
- Chain of custody records
- Custody seals
- Air sampling worksheets
- Sample labels
- Small screwdriver set
- Aluminum foil
- Extension cords
- Glass cracker
- Multiple plug outlet
- Whirl bags or culture tubes
- Teflon tape
- Calibration devices
- Tygon and/or Teflon^{R} tubing
- Surgical gloves
- Lint-free gloves
- Ice
- Sample container

Use the following additional equipment when decontaminating glassware on site:

- Protective equipment (i.e., gloves, splash goggles, etc.)
- Appropriate solvent(s)
- Spray bottles
- Liquinox (soap)
- Paper towels

- Distilled/deionized water
- Five-gallon buckets
- Scrub brushes and bottle brushes

6.0 REAGENTS

Impinger sampling involves using reagents contained in a glass vial to absorb contaminants of concern (for example, NIOSH Method 3500 for formaldehyde uses 1% sodium bisulfite solution). Impinger solutions vary and are method-dependent.

Reagents such as acetone and hexane are required to decontaminate glassware and some air sampling equipment. Decontamination solutions are specified in the Sampling Equipment Decontamination SOP.

7.0 PROCEDURES

7.1 Air Monitoring Design

7.1.1 Initial Surveys

In general, the initial survey is considered to be a relatively rapid screening process for collecting preliminary data at hazardous waste sites. However, initial surveys may require many hours to complete and may consist of more than one entry.

Some information is generally known about the site; therefore, real-time instrumentation for specific compounds (i.e., detector tubes and electrochemical sensors) can be used to identify hot spots. Sufficient data should be obtained with real-time instruments during the initial entry to screen the site for various contaminants. When warranted, intrinsically safe or explosion-proof instruments should be used. An organic vapor analyzer (OVA) is typically used during this survey. These gross measurements may be used on a preliminary basis to (1) determine levels of personal protection, (2) establish site work zones, and (3) map candidate areas for more thorough qualitative and quantitative studies involving air sampling.

In some situations, the information obtained may be sufficient to preclude additional monitoring. Materials detected during the initial survey may call for a more comprehensive evaluation of hazards and analyses for specific compounds. Since site activities and weather conditions change, a continuous program to monitor the ambient atmosphere must be established.

7.1.2 Off-Site Monitoring

Typically, perimeter monitoring with the same instruments employed for on-site monitoring is utilized to determine site boundaries. Because air is a dynamic matrix, physical boundaries like property lines and fences do not necessarily delineate the site boundary or area influenced by a release. Whenever possible, atmospheric hazards in the areas adjacent to the on-site zone should be monitored with direct-reading instruments. Monitoring at the fenceline or at varying locations off site provides useful information regarding pollutant migration. Three to four locations downwind of the source (i.e., plume) at breathing-zone height, provide a basic fingerprint of the plume. Negative instrument readings off site should not be interpreted as the complete absence of airborne toxic substances; rather, they should be considered another piece of information to assist in the preliminary evaluation. The interpretation of negative readings is instrument-dependent. The lack of instrument readings off site should not be interpreted as the complete absence of all airborne toxic substances; rather, it is possible that the particular compound or class of compounds to which the monitoring instrument responds is not present or that the concentration of the compound(s) is below the instrument's detection limit.

7.2 Air Sampling Design

7.2.1 Sampling Plan Design

The goal of air sampling is to accurately assess the impact of a contaminant source(s) on ambient air quality. This impact is expressed in terms of overall average and/or maximum air concentrations for the time period of concern and may be affected by the transport and release of pollutants from both on- and off-site sources. The location of these sources must be taken into account as they impact the selection of sampling locations. Unlike soil and groundwater concentrations, air concentrations at points of interest can easily vary by orders of magnitude over the period of concern. This variability plays a major role in designing an air sampling plan.

Downwind air concentration is determined by the amount of material being released from the site into the air (the emission rate) and by the degree that the contamination is diluted as it is transported. Local

meteorology and topography govern downwind dilution. Contaminant emission rates can also be heavily influenced by on-site meteorology and on-site activities. All of these concerns must be incorporated into an air sampling plan.

A sampling strategy can be simple or complex, depending on the sampling program objectives. Programs involving characterization of the pollutant contribution from a single point source tend to be simple, whereas sampling programs investigating fate and transport characteristics of components from diverse sources require a more complex sampling strategy. In addition, resource constraints may affect the complexity of the sampling design.

An optimal sampling strategy accounts for the following site parameters:

- Location of stationary as well as mobile sources
- Analytes of concern
- Analytical detection limit to be achieved
- Rate of release and transport of pollutants from sources
- Availability of space and utilities for operating sampling equipment
- Meteorological monitoring data
- Meteorological conditions in which sampling is to be conducted

The sampling strategy typically requires that the concentration of contaminants at the source or area of concern as well as background contributions be quantified. It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks, as well as various other types of QA/QC samples, can be utilized to determine other sources. The impact of extraneous sources on sampling results can frequently be accounted for by placing samplers upwind, downwind and crosswind from the subject source. The analytical data from these different sampling locations may be compared to determine statistical differences.

7.2.2 Sampling Objectives

The objectives of the sampling must be determined prior to developing the sampling plan. Does the sampling plan verify adequate levels of protection for on-site personnel, or address potential off-site impacts associated with the site or with site activities? In addition, the assumptions associated with the sampling program must be defined. These assumptions include whether the sampling is to take place under "typical," "worst case," or "one-time" conditions. If the conditions present at the time of sampling are different from those assumed during the development of the sampling plan, then quality of the data collected may be affected. The following definitions have been established:

- Typical: routine daily sampling or routine scheduled sampling at pre-established locations.
- Worst case: sampling conducted under the worst meteorological and/or site conditions which would result in elevated ambient concentrations.
- One-time: only one chance is given to collect a sample without regard to time or conditions.

Qualitative data acquired under these conditions are usually applicable only to the time period during which the data were collected and may not provide accurate information to be used in estimating the magnitude of an air impact during other periods or over a long time interval.

The sampling objectives also dictate the detection limits. Sampling methods for airborne contaminants will depend upon the nature and state (solid, liquid or gas) of the contaminant. Gases and vapors may be collected in aqueous media or adsorbents, in molecular sieves, or in suitable containers. Particulates are collected by filters or impactors. The volume of sample to be collected is dependent upon an estimate of the contaminant concentration in the air, the sensitivity of the analytical method, and the standard or desired detection limit. A sufficient amount of sample must be collected to achieve the desired detection limit without interference from other contaminants. In addition, the selected method must be able to detect the target compound(s).

7.2.3 Location and Number of Individual Sampling Points

Choose the number and location of sampling points according to the variability, or sensitivity, of the sampling and analytical methods being utilized, the variability of contaminant concentration over time at the site, the level of precision required and costlimitations. In addition, determine the number of locations and placement of samplers by considering the nature of the response, local terrain, meteorological conditions, location of the site (with respect to other conflicting background sources), size of the site, and the number, size, and relative proximity of separate on-site emission sources and upwind sources. The following are several considerations for sampler placement:

- Location of potential on-site emission sources, as identified from the review of site background information or from preliminary on-site inspections.

- Location of potential off-site emission sources upwind of the sampling location(s). Review local wind patterns to determine the location of off-site sources relative to wind direction.

- Topographic features that affect the dispersion and transport of airborne toxic constituents.

Avoid natural obstructions when choosing air sampling station locations, and account for channelization around those obstructions.

- Large water bodies, which affect atmospheric stability and the dispersion of air contaminants.

- Roadways (dirt or paved), which may generate dust that could mask site contaminants.

- Vegetation, such as trees and shrubs, which stabilizes soil and retards subsurface contaminants from becoming airborne. It also affects air flow and scrubs some contaminants from the air. Sometimes thick vegetation can make an otherwise ideal air monitoring location inaccessible.

Consider the duration of sampling activities when choosing the location and number of samples to be collected. For example, if the sampling period is limited to a few hours, one or two upwind and several downwind samples would typically be adequate, especially around major emission sources.

A short-term monitoring program ranges from several days to a few weeks and generally includes gathering data for site assessments, removal actions, and source determination data (for further modeling). Activities involved in a short-term sampling strategy must make the most of the limited possibilities for data collection. Consider moving upwind/downwind locations daily based on National Oceanic and Atmospheric Administration (NOAA) weather forecasts. Weather monitoring becomes critical where complex terrain and local meteorological effects frequently change wind direction. Often, a number of alternatives can fulfill the same objective.

Prevailing winds running the length of a valley usually require a minimum number of sampler locations; however, a complex valley may require more sampler locations to account for the wide variety of winds. Ocean/lake effects may require a radical plan to collect enough samples to reach a low detection limit. Two sets of samplers may be placed next to each other: one set would be activated during the sea breeze

while the other set is turned off, and vice versa when there is no sea breeze. After the sampling event, the respective upwind and downwind samples would be combined. Another alternative for sampling near a large body of water may be to use automatic, wind-vector-operated samplers, which turn the sampler on only when the wind comes from a specified vector. At sites located on hillsides, wind will move down a valley and produce an upward fetch at the same time. Sampling locations may have to ring the site to measure the wind's impact.

Off-site sources may affect on-site monitoring. In this case, on-site meteorological data, concurrent with sampling data, is essential to interpreting the acquired data. Also, additional upwind sampling sites may be needed to fully characterize ambient background contaminant levels. Multiple off-site sources may require several monitoring locations, but if the sources are at a sufficient distance, only one monitoring location is needed.

Topography and weather are not the only factors in sampler location; the sampling sites must be secure from vandals and mishap. Secure all sampling locations to maintain chain of custody, and to prevent tampering with samples or loss of sampling units. High-volume sampling methods often require the use of 110 VAC electric power. When portable generators are used, the power quality may affect sampler operation. Also, be aware that the generators themselves could be a potential pollution source if their placement is not carefully considered.

Air quality dispersion models can be used to place samplers. The models incorporate source information, surrounding topography, and meteorological data to predict the general distance and directions of maximum ambient concentrations. Modeling results should be used to select sampling locations in areas of maximum pollutant concentrations.

7.2.4 Time, Duration and Frequency of Sampling Events

After choosing appropriate sampling or monitoring locations, determine the sampling frequency and the number of samples to be collected. The time of day, duration and frequency of sampling events is governed by:

- The effects of site activities and meteorology on emission rates
- The diurnal effect of the meteorology on downwind dispersion
- The time period(s) of concern as defined by the objective
- The variability in the impact from other non-site-related sources
- If defined, the degree of confidence needed for either the mean or maximum downwind concentrations observed
- The precision requirements for single measurements
- Cost and other logistical considerations

The duration of the removal action and the number of hours per day that site work is conducted determine the time, duration, and frequency of samples. Short-term sampling programs may require daily sampling, while long-term programs may require 24-hour sampling every sixth or twelfth day. If the site will be undergoing removal activities 24 hours a day, continuous air sampling may be warranted. However, if the site activities will be conducted for only eight hours a day, and there are no emissions likely to occur during the remaining 16 hours, then sampling would be appropriate prior to the start of daily activities, would continue during operations, and end at the conclusion of the daily activities. An off-peak sample collection can ensure that emissions are not persisting after the conclusion of daily cleanup activities. For some sites, emissions are still a factor several hours after daily site activities have been completed. Because of the typically decreased downwind dispersion in the evening, higher downwind concentrations than were present during daytime site activities may be detected. For sites where this is possible, the sampling duration needs to be lengthened accordingly.

Sampling duration and flow rate dictate the volume of air collected, and to a major degree, the detection limit. The analytical method selected will provide a reference to flow rate and volume. Flow rates are limited to the capacity of the pumps being employed and the contact time required by the collection media.

The duration or period of air sampling is commonly divided into two categories (1) samples collected over a brief time period are referred to as "instantaneous" or "grab" samples and are usually collected in less than five minutes and (2) average or integrated samples are collected over a significantly longer period of time. Integrated samples provide an average

concentration over the entire sampling period. Integrated samples are not suited to determining cyclical releases of contaminants because periodic or cyclical events are averaged out by the proportionally long sampling duration.

Air quality dispersion models can predict the maximum air contaminant concentration expected from a source. The meteorological and site conditions expected to cause the highest concentration are known as worst-case conditions and can be identified by analyzing the modeling results. Depending upon the objective, one may sample when the model predicts worst-case conditions will exist.

7.2.5 Meteorological and Physical/Chemical Considerations

A meteorological monitoring program is an integral part of site monitoring activities. Meteorological data, which define local terrain impacts on air flow paths, are needed to interpret air concentration data. Meteorological data may be available from an existing station located near the site (i.e., at a local airport), otherwise a station should be set up at the site. This data will document the degree that samples actually were downwind and verify whether other worst-case assumptions were met. Meteorological parameters to be monitored are, at a minimum, wind speed, wind direction, and sigma theta (which is the horizontal wind direction standard deviation and an indicator of atmospheric stability). The remaining parameters primarily affect the amount of a contaminant available in the air.

- Wind Speed

  When the contaminant of concern is a particulate, wind speed is critical in determining whether the particulate will become airborne, the quantity of the particulate that becomes airborne, and the distance the particulate will travel from the source. Wind speed also contributes to the volatilization of contaminants from liquid sources.

- Wind Direction

  Wind direction highly influences the path of airborne contaminants. In addition, variations in wind direction increase the dispersion of pollutants from a given source.

- Atmospheric Stability

  Atmospheric stability refers to the degree to which the atmosphere tends to dampen vertical and horizontal motion. Stable atmospheric conditions (i.e., evenings) result in low dispersion, and unstable atmospheric conditions (i.e., hot sunny days) result in higher dispersion.

- Temperature

  Higher temperatures increase the rate of volatilization of organic and some inorganic compounds and affect the initial rise of gaseous or vapor contaminants. Therefore, worst-case emission of volatiles and semivolatiles occurs at the hottest time of day, or on the hottest day.

- Humidity

  High humidity affects water-soluble chemicals and particulates. Humid conditions may dictate the sampling media used to collect the air sample, or limit the volume of air sampled and thereby increase the detection limit.

- Atmospheric Pressure

  Migration of landfill gases through the landfill surface and through surrounding soils are governed by changes in atmospheric pressure. Atmospheric pressure will influence upward migration of gaseous contaminants from shallow aquifers into the basements of overlying structures.

In many cases, the transport and dispersion of air pollutants is complicated by local meteorology. Normal diurnal variations (i.e., temperature inversions) affect dispersion of airborne contaminants. Terrain features can enhance or create air inversions and can also influence the path and speed of air flow, complicating transport and dispersion patterns.

The chemical characteristics of a contaminant (i.e., molecular weight, physical

state, vapor pressure, aerodynamic size, temperature, reactive compounds, and photodegradation) affects its behavior and can influence the method used to sample and analyze it.

8.0 CALCULATIONS

Volume is obtained by multiplying the sample time in minutes by the flow rate. Sample volume should be indicated on the chain of custody record. Adjustments for temperature and pressure differences may be required.

Results are usually provided in parts per million (ppm), parts per billion (ppb), milligrams per cubic meter (mg/m^3) or micrograms per cubic meter ($\mu g/m^3$).

Refer to the analytical method or regulatory guidelines for other applicable calculations.

9.0 QUALITYASSURANCE/ QUALITY CONTROL

The manufacturer's instructions should be reviewed prior to instrument use. Instruments must be utilized in accordance with manufacturer's instructions. Equipment checkout and calibration activities must occur prior to and after monitoring and sampling and must be documented.

9.1 QA/QC Samples

QA/QC samples provide information on the variability and usability of environmental sample results. Various QA/QC samples may be collected to detect error. QA/QC samples are submitted with the field samples for analysis to aid in identifying the origin of analytical discrepancies; then a determination can be made as to how the analytical results should be used. Collocated samples, background samples, field blanks, and lot blanks are the most commonly collected QA/QC field samples. Performance evaluation (PE) samples and matrix spikes provide additional measures of data QA/QC control. QA/QC results may suggest the need for modifying sample collection, preparation, handling, or analytical procedures if the resultant data do not meet site-specific QA or data quality objectives.

9.2 Sample Documentation

All sample and monitoring activities should be documented legibly, in ink. Any corrections or revisions should be made by lining through the incorrect entry and by initialing the error. All samples must be recorded on an Air Sampling Worksheet. A chain of custody record must be maintained from the time a sample is taken to the final deposition of the sample. Custody seals demonstrate that a sample container has not been opened or tampered with during transport or storage of samples.

10.0 DATA VALIDATION

Results for QA/QC samples should be evaluated for contamination. This information should be utilized to qualify the environmental sample results accordingly with data quality objectives.

11.0 HEALTH AND SAFETY

Personal protection equipment (PPE) requirements identified in federal and/or state regulations and 29 Code of Federal Regulations (CFR) 1910.120 for hazardous waste site work must be followed.

The majority of physical precautions involved in air sampling are related to the contaminant sampled. Attention should be given when sampling in potentially explosive, flammable or acidic atmospheres. On rare occasions, the collection media may be hazardous; for example, in the instance where an acidic or basic solution is utilized in an impinger.

When working with potentially hazardous materials, follow U.S. EPA, OSHA and corporate health and safety procedures.

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APPENDIX A

PORTABLE SCREENING DEVICES

Where possible, a datalogger should be used to minimize the length of time required for site personnel to be in a potentially contaminated area. Datalogger cable is available from manufacturers for linear output instruments and some nonlinear output instruments. U.S. EPA ERT/REAC has output cables for organic vapor analyzers (i.e., HNU and OVA), toxic gas analyzers (i.e., monitox) and real-time aerosol monitors (i.e., RAM and miniram).

- Total Hydrocarbon Analyzers

  Total hydrocarbon analyzers used to detect a variety of volatile organic compounds (VOCs) at hazardous waste sites principally employ either a photoionization detector (PID) or a flame ionization detector (FID). Compounds are ionized by a flame or an ultraviolet lamp. PIDs depend on the ionization potential of the compounds. PIDs are sensitive to aromatic and olefinic (unsaturated) compounds such as benzene, toluene, styrene, xylenes, and acetylene. Greater selectivity is possible by using low-voltage lamps. The ionization potential of individual compounds can be found in the NIOSH Pocket Guide to Chemical Hazards. These instruments are not compound-specific and are typically used as screening instruments. FIDs are sensitive to volatile organic vapor compounds such as methane, propanol, benzene and toluene. They respond poorly to organic compounds lacking hydrocarbon characteristics.

- Oxygen and Combustible Gas Indicators

  Combustible Gas Indicators (CGIs) provide efficient and reliable methods to test for potentially explosive atmospheres. CGI meters measure the concentration of a flammable vapor or gas in air and present these measurements as a percentage of the lower explosive limit (LEL).

The measurements are temperature-dependent. The property of the calibration gas determines sensitivity. LELs for individual compounds can be found in the NIOSH Pocket Guide to Chemical Hazards. If readings approach or exceed 10% of the LEL, extreme caution should be exercised in continuing the investigation. If readings approach or exceed 25% LEL, personnel should be withdrawn immediately.

CGIs typically house an electrochemical sensor to determine the oxygen concentration in ambient air. Normally, air contains approximately 20.9% oxygen by volume. Oxygen measurements are of particular importance for work in enclosed spaces, low-lying areas, or in the vicinity of accidents that have produced heavier-than-air vapors which could displace ambient air. The meters are calibrated for sea level and may indicate a false negative (i.e, O_2 content) at higher altitudes. Since the air has been displaced by other substances, these oxygen-deficient areas are also prime locations for taking additional organic vapor and combustible gas measurements. Oxygen-enriched atmospheres increase the potential for fires by their ability to contribute to combustion or to chemically react with flammable compounds and promote auto-ignition.

- Toxic Atmosphere Analyzers

  The toxic atmosphere analyzer is a compound-specific instrument, designed and calibrated to identify and quantify a specific compound or class of compounds in either gaseous or vapor form. Cross-sensitivity to air pollutants not of interest may be lead to erroneous results.

  U.S. EPA/ERT has the following toxic atmosphere analyzers: carbon monoxide, phosgene, nitrous oxide, hydrogen cyanide, sulfur dioxide, hydrogen sulfide, and chlorine gas.

- Aerosol/Particulate Monitors

  A Real-Time Aerosol/Particulate Monitor (RAM) displays readings for total particulates. The instrument employs a pulse light emitting diode which generates a narrow band emission in conjunction with a photovoltaic cell to detect light scattered from particulates.

  The U.S. EPA/ERT uses the RAM when the contaminant of concern is associated with particulates, and when responding to fires involving hazardous materials, to identify plume levels. The instrument is very useful in determining the presence of a plume when it is not visible. The U.S. EPA/ERT typically uses RAMs on tripods to obtain particulate concentrations at the breathing zone level. Personal dataloggers are used with the RAMs to document minimum, average and maximum concentrations. This provides real-time data without requiring those in personal protective equipment to be constantly present in the plume.

- Chemical Detector Tubes (Colorimetric Tubes)

  A chemical detector tube is a hollow, tube-shaped, glass body containing one or more layers of chemically impregnated inert material. To use, the fused ends are broken off and a manufacturer-specified volume of air is drawn through the tube with a pump to achieve a given detection limit. The chemicals contained within the packing material undergo a chemical reaction with the airborne pollutant present, producing a color change during the intake of each pump stroke. The concentration of a pollutant is indicated by the length of discoloration on a calibrated scale printed on the detector tube.

- Radiation Meters

  Radiation meters determine the presence and level of radiation. The meters use a gas or solid ion detection media which becomes ionized when radiation is present. The meters are normally calibrated to one probe. Meters that detect alpha, beta, and gamma radiation are available.

- Gold Film (Hydrogen Sulfide and Mercury Vapor) Monitors

  Hydrogen sulfide (H_2S) and Mercury (Hg) monitors operate on the principle that electric resistivity increases across a gold film as a function of H_2S and Hg concentration. The monitors provide rapid and relatively low detection limits for H_2S and Hg in air. After extensive sampling periods or high concentrations of H_2S and Hg, the gold film must be heated to remove contamination and return the monitor to its original sensitivity.

- Infrared Detectors

  Infrared detectors such as the Miniature Infrared Analyzer (MIRAN) use infrared (IR) absorption as a function of specific compounds. MIRAN instruments apply to situations where the contaminants are identified but concentrations are not. MIRAN instruments generally require AC power.

SPECIALIZED ANALYTICAL INSTRUMENTS

The continuous monitors described above provide qualitative measurement of air contaminants. Quantitative measurements in the field can be obtained using more sophisticated instruments, such as portable Gas Chromatographs, to analyze grab samples.

- Direct Air Sampling Portable Gas Chromatographs (GCs)

  Portable GCs use gas chromatography to identify and quantify compounds. The time it takes for a compound to move through a chromatographic column is a function of that specific compound or group of compounds. A trained technician with knowledge of the range of expected concentrations of compounds can utilize a portable GC in the field to analyze grab samples. GCs generally require AC power and shelter to operate. This method is limited by its reliance on a short-term grab sample to be representative of the air quality at a site.

- Remote Optical Sensing

  This technique, also referred to as long-path or open-path monitoring, involves transmitting either an infrared or ultraviolet light beam across a long open path and measuring the absorbance at specific wavelengths. The technique is capable of analyzing any preselected organic or inorganic volatile compound that can be resolved from compounds naturally occurring in ambient air. Current projected removal applications include perimeter monitoring during site cleanups and measurement of emission source strengths during site assessments.

- TAGA Direct Air Sampling Mass Spectrometer/Mass Spectrometer

  The Trace Atmospheric Gas Analyzer (TAGA), which is operated by the U.S. EPA/ERT, is capable of real-time detection of preselected organic compounds at low parts-per-billion concentrations. The instrument has been successfully used by the U.S. EPA/ERT for isolating individual emission plumes and tracking those plumes back to their sources.

APPENDIX B

Air Sampling Equipment and Media/Devices

AIR SAMPLING EQUIPMENT

- High-Volume, Total Suspended Particulate (TSP) Samplers

  High-volume TSP samplers collect all suspended particles by drawing air across an 8- by 10-inch glass-quartz filter. The sample rate is adjusted to 40 cubic feet per minute (CFM), or 1134 liters per minute (L/min), and it is held constant by a flow controller over the sample period. The mass of TSPs can be determined by weighing the filter before and after sampling. The composition of the filter varies according to the analytical method and the detection limit required.

- PM-10 Samplers

  PM-10 samplers collect particulates with a diameter of 10 microns or less from ambient air. Particulates of this size represent the respirable fraction, and thus are of special significance. PM-10 samplers can be high-volume or low-volume. The high-volume sampler operates in the same manner as the TSP sampler at a constant flow rate of 40 CFM; it draws the sample through a special impactor head which collects particulates of 10 microns or less. The particulate is collected on an 8- by 10-inch filter. The low-volume sampler operates at a rate of approximately 17 L/min. The flow must remain constant through the impactor head to maintain the 10-micron cut-off point. The low-volume PM-10 collects the sample on 37-mm Teflon filters.

- High-Volume PS-1 Samplers

  High-volume PS-1 samplers draw a sample through polyurethane foam (PUF) or a combination foam and XAD-2 resin plug, and a glass quartz filter at a rate of 5-10 CFM (144 to 282 L/min). This system is excellent for measuring low concentrations of semivolatiles, PCBs, pesticides, or chlorinated dioxins in ambient air.

- Area Sampling Pumps

  These pumps provide flow-rate ranges of 2-20 L/min and have a telescopic sampling mast with the sampling train. Because of the higher volume, this pump is suitable for sampling low concentrations of airborne contaminants (i.e., asbestos sampling). These pumps are also used for metals, pesticides and PAH sampling which require large sample volumes.

- Personal Sampling Pumps

  Personal sampling pumps are reliable portable sampling devices that draw air samples through a number of sampling media including resin tubes, impingers, and filters. Flow rates are usually adjustable from 0.1 to 4 L/min (or 0.01 to .75 L/min with a restrictive orifice) and can remain constant for up to 8 hours on one battery charge or continuously with an AC charger/converter.

- Canister Samplers

  Evacuated canister sampling systems use the pressure differential between the evacuated canister and ambient pressure to bleed air into the canister. The sample is bled into the canister at a constant rate over the sampling period using a critical orifice, a mechanically compensated regulator, or a mass flow control
  device until the canister is near atmospheric pressure.

  Pressure canister sampling systems use a pump to push air into the canister. To maintain a higher, more controlled flow, the pump typically controls the pressure differential across a critical orifice at the

inlet of the canister, resulting in a pressurized canister at the completion of sampling.

AIR SAMPLING MEDIA/DEVICES

If possible, before employing a specific sampling method, consult the laboratory that will conduct the analyses. Many of the methods can be modified to provide better results or a wider range of results.

- Summa[R] Canisters

  Summa canisters are highly polished passivated stainless steel cylinders. The Summa polishing process brings chrome and nickel to the surface of the canisters, which results in an inert surface. This surface restricts adsorption or reactions that occur on the canister's inner surface after collection. At the site, the canister is either placed in a sampler to control sample collection rate, or opened to collect a grab sample. Samples can be collected by allowing air to bleed into or be pumped into the canister. U.S. EPA/ERT uses 6-liter Summa canisters for VOC and permanent gas analysis.

- Passive Dosimeters

  Passive dosimeters are clip-on vapor monitors (samplers) in which the diffused contaminants are absorbed on specially prepared active surfaces. Industrial hygienists commonly use dosimeters to obtain time-weighted averages or concentrations of chemical vapors, as they can trap over 130 organic compounds. Selective dosimeters have also been developed for a number of chemicals including formaldehyde, ethylene oxide, hydrogen sulfide, mercury vapor, nitrogen dioxide, sulfur dioxide, and ozone. Dosimeters must be sent to a laboratory for analysis.

- Polyurethane Foam (PUF)

  PUF is a sorbent used with a glass filter for the collection of semivolatile organic compounds such as pesticides, PCBs, chlorinated dioxins and furans, and PAHs. Fewer artifacts (chemical changes that occur to collected compounds) are produced than with some other solid sorbents. PUF is used with the PS-1 sampler and U.S. EPA Method TO13. PUF can also be used with personal sampling pumps when sampling for PAHs using the Lewis/McCloud method. Breakthrough of the more volatile PCBs and PAHs may occur when using PUF.

- Sampling Bags (Tedlar[R])

  Sampling bags, like canisters, transport air samples to the laboratory for analysis. Samples are generally pumped into the bags, but sometimes a lung system is used, in which a pump creates a vacuum around the bag in a vacuum box. Then the sample flows from a source into the bag. This method is used for VOCs, fixed gases (CO_2, O_2, and N_2) and methane.

- Impingers

  An impinger allows an air sample to be bubbled through a solution, which collects a specific contaminant by either chemical reaction or absorption. For long sampling periods, the impinger may need to be kept in an ice bath to prevent the solution from evaporating during sampling. The sample is drawn through the impinger by using a sampling pump or more elaborate sampling trains with multiple impingers.

- Sorbent Tubes/Cartridges

  A variety of sampling media are available in sorbent tubes, which are used primarily for industrial hygiene. A few examples are carbon cartridges, carbon molecular sieves, Tenax tubes and tube containing the XAD-2 polymer. Depending upon the sorbent material, tubes can be analyzed using either a solvent extraction or thermal desorption. The former technique uses standard laboratory equipment and allows for multiple analyses of the same sample. The latter technique requires special, but readily available, laboratory equipment and allows only one analysis per sample. In addition, thermal desorption typically allows for lower detection limits by two or more orders of magnitude. Whenever sorbent tubes are

being used for thermal desorption, they should be certified as "clean" by the laboratory doing the analysis.

Thermally Desorbed Media

During thermal desorption, high-temperature gas streams are used to remove the compounds collected on a sorbent medium. The gas stream is injected and often cryofocused into an analytical instrument, such as a GC, for compound analysis:

- Tenax Tubes

  Tenax tubes are made from commercially available polymer (p-phenylene oxide) packed in glass or stainless steel tubes through which air samples are drawn or sometimes pumped. These tubes are used in U.S. EPA Method TO1 and VOST for volatile nonpolar organic, some polar organic, and some of the more volatile semivolatile organics. Tenax is not appropriate for many of the highly volatile organics (with vapor pressure greater than approximately 200 mm Hg).

- Carbonized Polymers

  The carbonized molecular sieve (CMS), a carbonized polymer, is a commercially available, carbon sorbent packed in stainless-steel sampling tubes through which air samples are drawn or sometimes pumped. These are used in U.S. EPA Method TO2 for highly volatile nonpolar compounds which have low-breakthrough volumes on other sorbents. When high-thermal desorption temperatures are used with CMS, more variability in analysis may occur than with other sorbents.

- Mixed Sorbent Tubes

  Sorbent tubes can contain two type of sorbents. Combining the advantages of each sorbent into one tube increases the possible types of compounds to be sampled. The combination of two sorbents can also reduce the chance that highly volatile compounds will break through the sorbent media. An example of a mixed sorbent tube is the combination of Tenax and charcoal with a carbonized molecular sieve. A potential problem with mixed sorbent tubes is the breakthrough of a compound from an earlier sorbent to a later sorbent from which it

cannot be desorbed.

Solvent-Extracted Media

Solvent-extracted media use the principle of chemical extraction to remove compounds collected on a sorbent media. The chemical solvent is injected into an instrument, such as a GC, for analysis of compounds. Examples of solvent-extracted media follow:

- Chemically Treated Silica Gel

  Silica gel is a sorbent which can be treated with various chemicals. The chemically treated silica gel can then be used to sample for specific compounds in air. Examples include the DNPH-coated silica gel cartridge used with U.S. EPA Method TO11.

- XAD-2 Polymers

  XAD-2 polymers usually are placed in tubes, custom-packed sandwich-style with polyurethane foam, and prepared for use with U.S. EPA Method TO13 or the semi-VOST method. The polymers are used for the collection of semivolatile polar and nonpolar organic compounds. The compounds collected on the XAD-2 polymer are chemically extracted for analysis.

- Charcoal Cartridges

  Charcoal cartridges, consisting of primary and backup sections, trap compounds by adsorption. Ambient air is drawn through them so that the backup section verifies that breakthrough of the analytes on the first section did not occur, and the sample collection was therefore quantitative. Quantitative sample collection is evident by the presence of target chemicals on the first charcoal section and the absence on the second section. Next, the adsorbed compounds must be eluted, usually with a solvent extraction, and analyzed by GC with a detector, such as a Mass Spectrometer (MS).

- Tenax Tubes

  Cartridges are used in OSHA and NIOSH methods in a manner similar to charcoal cartridges but typically for less volatile

compounds.

Particulate Filters

Particulate filters are used by having a sampling pump pass air through them. The filter collects the particulates present in the air and is then analyzed for particulate mass or chemical or radiological composition. Particulate filters are made from different materials which are described below.

- Mixed Cellulose Ester (MCE)

  MCE is manufactured from mixed esters of cellulose which are a blend of nitro-cellulose and cellulose acetate. MCE filters are used often for particulate sampling.

- Glass Fiber

  Glass fiber is manufactured from glass fibers without a binder. Particulate filters with glass fiber provide high flow rates, wet strength, and high, solid holding capacity. Generally, the filters are used for gravimetric analysis of particulates.

- Polyvinyl Chloride

  Particulate filters with polyvinyl chloride are resistant to concentrated acids and alkalis. Their low moisture pickup and light tare weight make them ideal for gravimetric analysis.

- Teflon

  Teflon is manufactured from polytetrafluorethylene (PTFE). Particulate filters with Teflon are easy to handle and exceptionally durable. Teflon filters are used for metal collection.

- Silver

  Particulate filters manufactured from pure silver have high collection efficiency and uniform pore size. These filters are used for mercury collection and analysis.

- Cellulose

  Particulate filters with cellulose contain less than 0.01% ash. These filters are used to collect particulates.

ATTACHMENT C

Air Sampling Worksheet

BOSSERT
AIR QUALITY INVESTIGATION
Sheet No._____

FIELD SAMPLE DATA SHEET FOR STATIONARY AIR

Address or Location ID:_____
Use Category: Residential Commerical Other (_____)

Site Visit Date:_____ **Sampling Team:**_____

Sample ID			
Location/Desc.			
Category (circle)	Sample Duplicate_____ Field Blank Lot Blank	Sample Duplicate_____ Field Blank Lot Blank	Sample Duplicate_____ Field Blank Lot Blank
Analysis			
Sample Media			
Flow Meter Type			
Flow Meter ID No.			
Pump ID No.			
Flow Rate: (Start/Final)			
Flow Rate: (Average)			
Time: (Start/Finish)			
Total Elapsed Time (min.)			
Calculated: Sample Volume (L)			
Pump Fault: (Y/N)			

Comments/Weather

Field Comments